Supporting Information

Hydroxyamination of aryl C−H bonds with *N*-hydroxycarbamate by synergistic Rh/Cu catalysis at room temperature†

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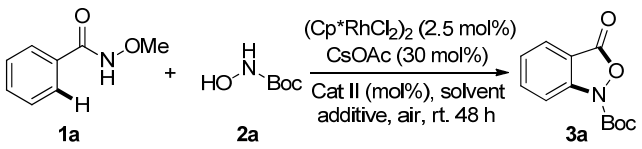
I. General Information

All Rhodium-catalyzed reactions were carried out without any particular precautions to extrude moisture or oxygen.

All reagents were purchased from commercial sources and used without further purification, unless otherwise indicated. All reactions were monitored by TLC, which was performed on precoated aluminum sheets of silica gel 60 (F254). The products were purified by flash column chromatography on silica gel (300−400 mesh). Melting points were uncorrected. NMR spectra were obtained on a Varian Inova 500 spectrometer (500 MHz for ^{1}H NMR; 125 MHz for ^{13}C NMR), with TMS as the internal standard. All chemical shifts are given in ppm. High-resolution mass spectra (HRMS) were obtained using a Bruker microTOF II focus spectrometer (ESI). GC/MS spectra were obtained on a Agilent 6890/5975 spectrometer (EI).

The starting materials were prepared according to the literature procedures.[1]

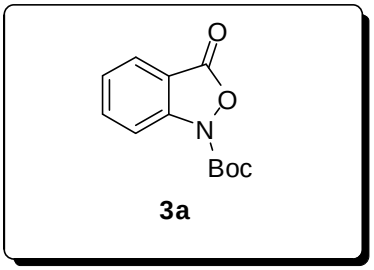
II. Typical Procedures and Analytical Data of 3a-3s



General procedure for the synthesis of 3a-3s (**3a** as an example):

Without any particular precautions to extrude oxygen or moisture, to a stirred mixture of **1a** (30.2 mg, 0.2 mmol) and **2a** (26.6 mg, 0.2 mmol) in EtOH (1.0 mL)/acetone (1.0 mL), $[Cp^*RhCl_2]_2$ (3.1 mg, 0.005 mmol), CsOAc (11.5 mg, 0.06 mmol), CuCl (2.0 mg, 0.02 mmol) and PivOH (20.4 mg, 0.2 mmol) were added successively, the reaction mixture was stirred at room temperature for 24 h. Then the other portion of **2a** (26.6 mg, 0.2 mmol) was added to the reaction mixture and stirred for another 24 h. The reaction mixture was diluted with brine (10 mL) and extracted with CH_2Cl_2 (2 × 10 mL). The combined organics were dried (Na_2SO_4), and concentrated *in vacuo*. The residue was purified by column chromatography (petroleum ether/EtOAc = 30/1, v/v) to afford the desired product **3a**.

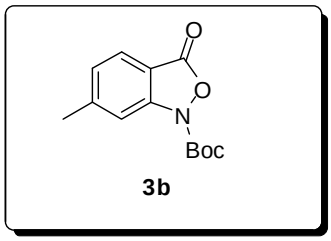
***tert*-Butyl 3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3a)**



White solid, m.p. 144–145 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.65 (s, 9H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.82 (d, *J* = 8.5 Hz, 1H), 7.88 (d, *J* = 8.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 86.0, 111.5, 113.9, 124.9, 125.5, 136.1, 146.3, 147.5, 164.1. **HRMS**

(ESI-TOF) Calcd for ($C_{12}H_{13}NNaO_4^+$ [MNa]$^+$) 258.0737. Found 258.0726.

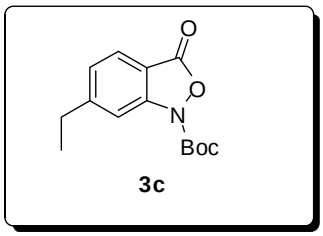
***tert*-Butyl 6-methyl-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3b)**



3b

White solid, m.p. 98−99 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.64 (s, 9H), 2.52 (s, 3H), 7.15 (d, *J* = 8.0Hz, 1H), 7.65 (s, 1H), 7.74 (d, *J* = 8.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 22.5, 28.1, 85.7, 109.0, 113.7, 125.1, 126.4, 146.7, 147.6, 148.0, 164.1. **HRMS** (ESI-TOF) Calcd for ($C_{13}H_{15}NNaO_4^+$ [MNa]$^+$) 272.0893. Found 272.0903.

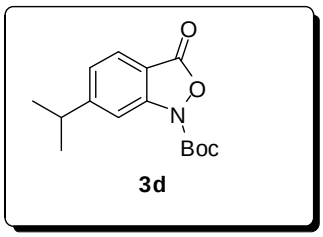
***tert*-Butyl 6-ethyl-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3c)**



3c

White solid, m.p. 106−108 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.31 (t, *J* = 7.5 Hz, 3H), 1.65 (s, 9H), 2.81 (q, *J* = 7.5 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.67 (s, 1H), 7.76 (d, *J* = 8.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 15.1, 28.1, 29.7, 85.8, 109.2, 112.6, 125.3, 125.5, 146.8, 147.6, 154.2, 164.1. **HRMS** (ESI-TOF) Calcd for($C_{14}H_{17}NNaO_4^+$ [MNa]$^+$) 286.1050. Found 286.1040.

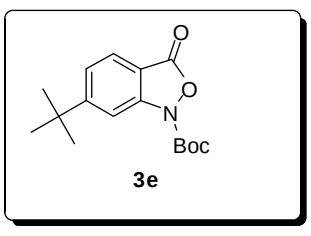
***tert*-Butyl 6-isopropyl-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3d)**



3d

White solid, m.p. 87−88 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.32 (d, *J* = 6.5 Hz, 6H), 1.65 (s, 9H), 3.06 (q, *J* = 6.5 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.69 (s, 1H), 7.78 (d, *J* = 8.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 23.6, 28.1, 35.1, 85.7, 109.3, 111.3, 124.2, 125.3, 146.8, 147.6, 158.9, 164.1. **HRMS** (ESI-TOF) Calcd for ($C_{15}H_{19}NNaO_4^+$ [MNa]$^+$) 300.1206. Found 300.1216.

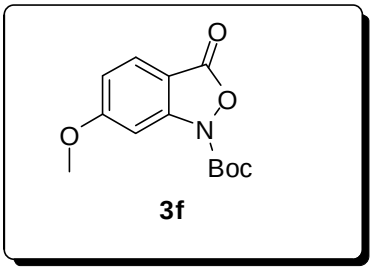
***tert*-Butyl 6-tert-butyl-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3e)**



3e

White solid, m.p. 109−110 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.39 (s, 9H), 1.66 (s, 9H), 7.41 (d, *J* = 8.0Hz, 1H), 7.78 (d, *J* = 8.5 Hz, 1H), 7.83 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 31.1, 36.0, 85.6, 108.9, 110.4, 123.2, 124.9, 146.8, 147.5, 161.1, 164.1. **HRMS** (ESI-TOF) Calcd for ($C_{16}H_{21}NNaO_4^+$ [MNa]$^+$) 314.1363. Found 314.1371.

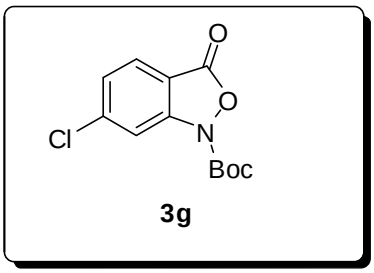
***tert*-Butyl 6-methoxy-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3f)**



3f

White solid, m.p. 139−140 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.64 (s, 9H), 3.93 (s, 3H), 6.87 (dd, *J* =7.0, 8.5 Hz, 1H), 7.27 (d, *J* =7.0Hz, 1H), 7.72 (d, *J* =8.5Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 56.0, 85.8, 96.2, 103.8, 114.9, 126.6, 147.5, 148.6, 163.8, 166.4. **HRMS** (ESI-TOF) Calcd for ($C_{13}H_{15}NNaO_5^+$ [MNa]$^+$) 288.0842. Found 288.0844.

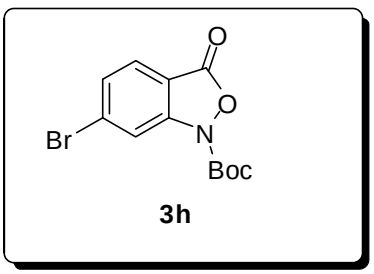
***tert*-Butyl 6-chloro-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3g)**



3g

White solid, m.p. 102−103 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.65 (s, 9H), 7.31 (d, *J* = 8.5Hz, 1H), 7.79 (d, *J* =8.5 Hz, 1H), 7.88 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 86.6, 109.9, 114.1, 125.8, 126.6, 143.0, 146.7, 147.1, 163.2. **HRMS** (ESI-TOF) Calcd for ($C_{12}H_{12}ClNNaO_4^+$ [MNa]$^+$) 292.0347. Found 292.0362.

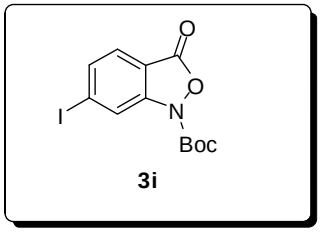
***tert*-Butyl 6-bromo-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3h)**



3h

White solid, m.p. 97−98 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.65 (s, 9H), 7.47 (d, *J* = 8.0Hz, 1H), 7.72 (d, *J* =8.5 Hz, 1H), 8.06 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 86.6, 110.3, 117.1, 126.6, 128.6, 131.5, 146.8, 147.1, 163.3. **HRMS** (ESI-TOF) Calcd for ($C_{12}H_{12}BrNNaO_4^+$ [MNa]$^+$) 335.9842. Found 335.9832.

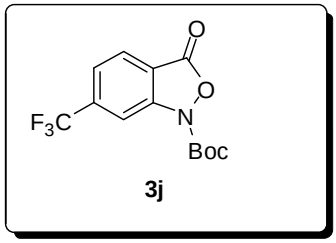
***tert*-Butyl 6-iodo-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3i)**



3i

White solid, m.p. 138−139 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.65 (s, 9H), 7.57 (d, *J* = 8.0Hz, 1H), 7.68 (d, *J* =8.0 Hz, 1H), 8.30 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.0, 86.5, 104.1, 110.9, 122.9, 126.4, 134.3, 146.5, 147.1, 163.5. **HRMS** (ESI-TOF) Calcd for ($C_{12}H_{12}INNaO_4^+$ $[MNa]^+$)383.9703. Found383.9711.

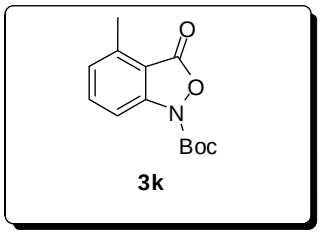
***tert*-Butyl 3-oxo-6-(trifluoromethyl)benzo[*c*]isoxazole-1(3*H*)-carboxylate (3j)**



3j

Yellow oil. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.66 (s, 9H), 7.59 (d, *J* = 8.5Hz, 1H), 8.02 (d, *J* =8.0 Hz, 1H), 8.16 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.0, 87.0, 111.7 (q, *J* = 4.3 Hz), 114.0, 121.6 (q, *J* = 3.4 Hz), 123.0 (q, *J* = 272.3 Hz), 126.6, 137.8 (q, *J* = 32.9 Hz), 145.9, 147.0, 162.9. **HRMS** (ESI-TOF) Calcd for ($C_{13}H_{12}F_3NNaO_4^+$ $[MNa]^+$) 326.0611. Found 326.0618.

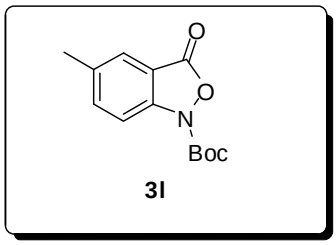
***tert*-Butyl 4-methyl-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3k)**



3k

White solid, m.p. 127-128 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.64 (s, 9H), 2.68 (s, 3H), 7.08 (d, *J* = 7.0Hz, 1H), 7.57-7.62 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 17.2, 28.1, 85.7, 109.7, 111.1, 125.9, 135.9, 139.8, 146.6, 147.5, 164.2. **HRMS** (ESI-TOF) Calcd for ($C_{13}H_{15}NNaO_4^+$ $[MNa]^+$) 272.0893. Found 272.0898

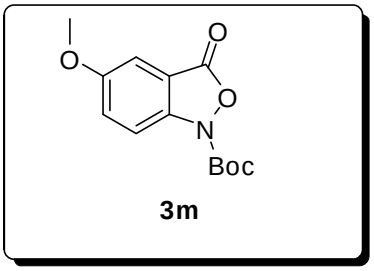
***tert*-Butyl 5-methyl-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3l)**



3l

White solid, m.p. 97−98 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.64 (s, 9H), 2.45 (s, 3H), 7.56 (d, *J* = 8.5Hz, 1H), 7.64 (s, 1H), 7.69 (d, *J* = 8.5Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 20.9, 28.1, 85.8, 111.6, 113.6, 124.8, 135.1, 137.5, 144.6, 147.6, 164.3. **HRMS** (ESI-TOF) Calcd for ($C_{13}H_{15}NNaO_4^+$ [MNa]$^+$) 272.0893. Found 272.0915.

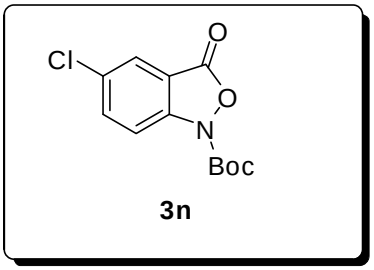
***tert*-Butyl 5-methoxy-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3m)**



3m

White solid, m.p. 144−145 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.64 (s, 9H), 3.86 (s, 3H), 7.23 (s, 1H), 7.35 (d, *J* = 8.5Hz, 1H), 7.71 (d, *J* = 9.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 55.9, 85.7, 105.2, 112.1, 115.2, 126.2, 141.3, 147.7, 157.2, 164.3. **HRMS** (ESI-TOF) Calcd for ($C_{13}H_{15}NNaO_5^+$ [MNa]$^+$) 288.0842. Found 288.0873.

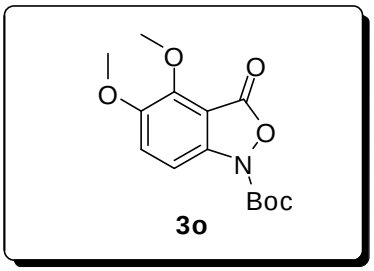
***tert*-Butyl 5-chloro-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3n)**



3n

White solid, m.p. 74−75 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.64 (s, 9H), 7.70 (dd, *J* = 2.0, 9.0Hz, 1H), 7.79 (d, *J* = 9.0Hz, 1H), 7.83 (d, *J* = 1.5Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 86.5, 112.8, 115.2, 124.9, 130.6, 136.5, 144.8, 147.2, 162.8. **HRMS** (ESI-TOF) Calcd for ($C_{12}H_{12}ClNNaO_4^+$ [MNa]$^+$) 292.0347. Found 292.0340.

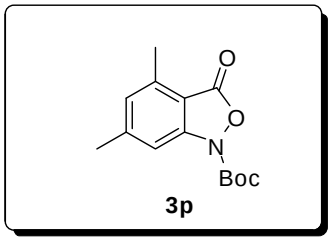
***tert*-Butyl 4,5-dimethoxy-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3o)**



3o

White solid, m.p. 97−98 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.63 (s, 9H), 3.91 (s, 3H), 4.16 (s, 3H), 7.35 (d, *J* = 9.0Hz, 1H), 7.43 (d, *J* = 9.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 57.6, 62.5, 85.6, 105.1, 107.7, 123.1, 141.3, 147.5, 148.0, 148.2, 162.0. **HRMS** (ESI-TOF) Calcd for ($C_{14}H_{17}NNaO_6^+$ [MNa]$^+$)318.0948. Found 318.0926.

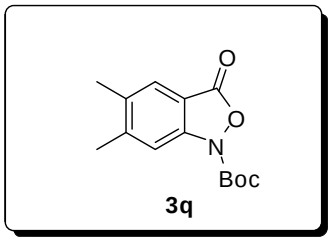
***tert*-Butyl 4,6-dimethyl-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3p)**



3p

White solid, m.p. 106−107 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.64 (s, 9H), 2.34 (s, 3H), 2.41 (s, 3H), 7.59 (s, 1H), 7.62 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 17.2, 22.4, 28.1, 85.6, 107.4, 111.1, 127.5, 139.3, 147.0, 147.7, 147.7, 164.2. **HRMS** (ESI-TOF) Calcd for ($C_{14}H_{17}NNaO_4^+$ $[MNa]^+$) 286.1050. Found 286.1048.

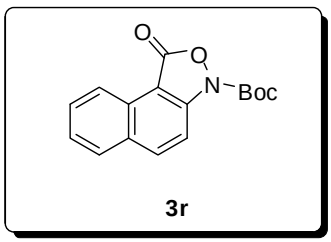
***tert*-Butyl 5,6-dimethyl-3-oxobenzo[*c*]isoxazole-1(3*H*)-carboxylate (3q)**



3q

White solid, m.p. 144−145 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.63 (s, 9H), 2.45 (s, 3H), 2.62 (s, 3H), 6.90 (s, 1H), 7.45 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 19.7, 21.3, 28.1, 85.6, 109.3, 114.2, 125.0, 134.4, 145.3, 147.2, 147.8, 164.3. **HRMS** (ESI-TOF) Calcd for ($C_{14}H_{17}NNaO_4^+$ $[MNa]^+$) 286.1050. Found 286.1050.

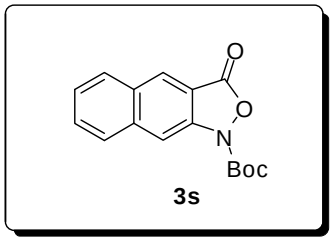
***tert*-Butyl 3-oxonaphtho[2,3-*c*]isoxazole-1(3*H*)-carboxylate (3r)**



3r

White solid, m.p. 164−165 °C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.68 (s, 9H), 7.59 (t, *J* = 7.5 Hz, 1H), 7.74 (t, *J* = 7.5 Hz, 1H), 7.93 (d, J = 8.5 Hz, 1H), 7.96 (d, *J* = 9.0 Hz, 1H), 8.16 (d, *J* = 9.0 Hz, 1H), 8.75 (d, *J* = 8.0Hz, 1H) . **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.1, 86.3, 103.4, 112.5, 122.7, 126.4, 128.5, 128.6, 129.9, 130.1, 137.9, 147.0, 147.1, 164.1. **HRMS** (ESI-TOF) Calcd for ($C_{16}H_{15}NNaO_4^+$ $[MNa]^+$) 308.0893. Found 308.0895.

***tert*-Butyl 1-oxonaphtho[2,1-*c*]isoxazole-3(1*H*)-carboxylate (3s)**

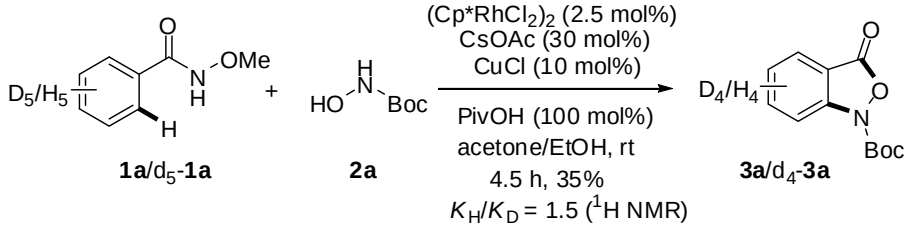


3s

White solid, m.p. 177−178°C. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.68 (s, 9H), 7.52 (t, *J* = 7.5Hz, 1H), 7.65 (t, *J* = 7.5Hz, 1H), 7.93 (d, *J* = 8.5Hz, 1H), 7.99 (d, *J* = 8.0Hz, 1H), 8.17 (s, 1H), 8.48 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$): δ 28.2, 85.7, 110.3, 112.2, 126.0, 127.4, 128.2, 129.8, 130.3, 137.6, 140.7, 148.4, 164.2. **HRMS** (ESI-TOF) Calcd for ($C_{16}H_{15}NNaO_4^+$ [MNa] $^+$) 308.0893. Found 308.0923.

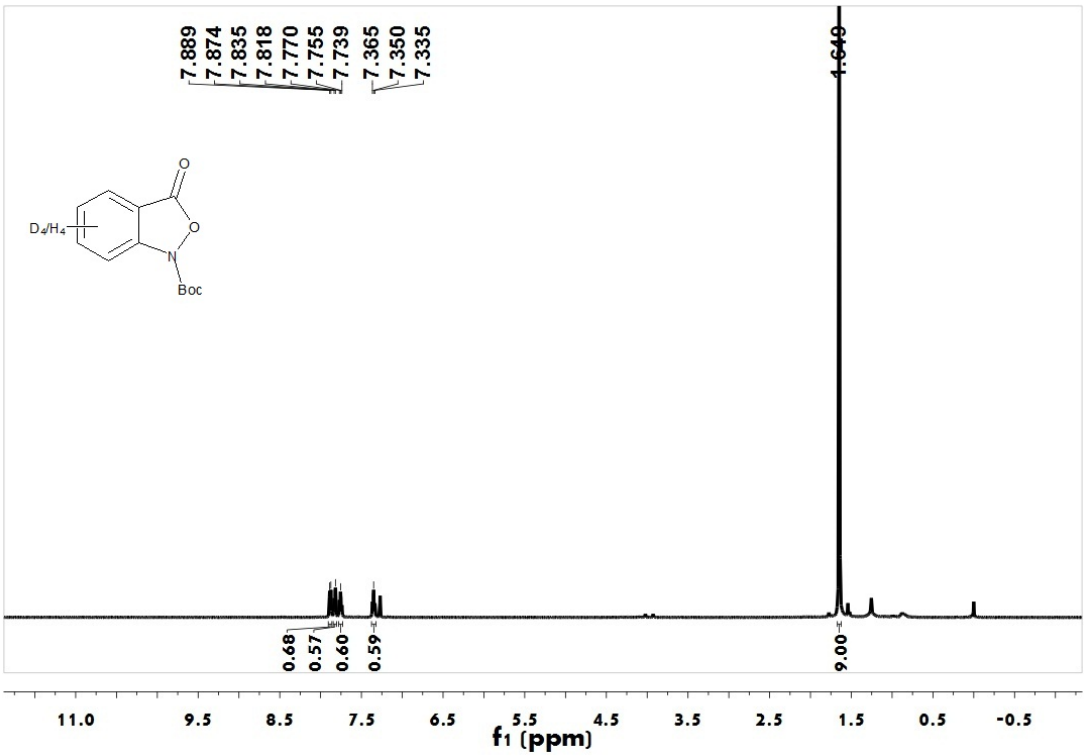
III. Mechanism experiments

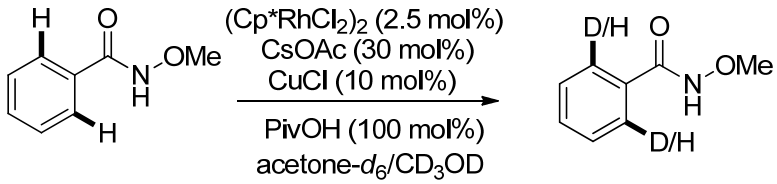
Interemolecular isotopic reaction:



1a/d_5-**1a** **2a** **3a**/d_4-**3a**

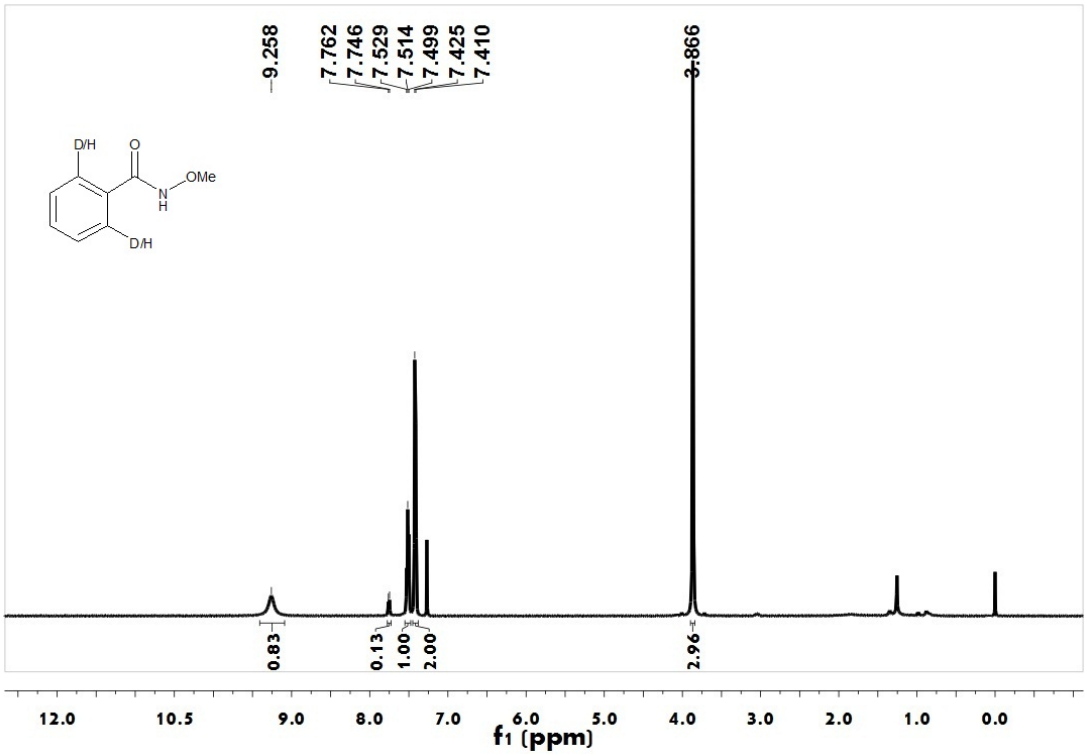
According to the general procedure, to a stirred mixture of **1a** (30.2 mg, 0.2 mmol), d_5-**1a** (31.2 mg, 0.2 mmol), and **2a** (26.6 mg, 0.2 mmol) in EtOH (1.0 mL)/acetone (1.0 mL), $[Cp^*RhCl_2]_2$ (3.1 mg, 0.005 mmol), CsOAc (11.5 mg, 0.06 mmol), CuCl (2.0 mg, 0.02 mmol) and PivOH (20.4 mg, 0.2 mmol) were added successively, the reaction mixture was stirred at room temperature for 4.5 h. The reaction mixture was diluted with brine (10 mL) and extracted with CH_2Cl_2 (2 × 10 mL). The combined organics were dried (Na_2SO_4), and concentrated *in vacuo*. The residue was purified by column chromatography (petroleum ether/EtOAc = 30/1, v/v) to afford the desired product **3a** and d_4-**3a** yield 35%. **KIE=1.5.**

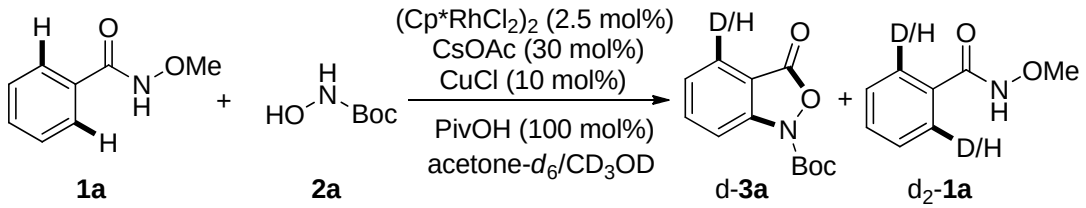




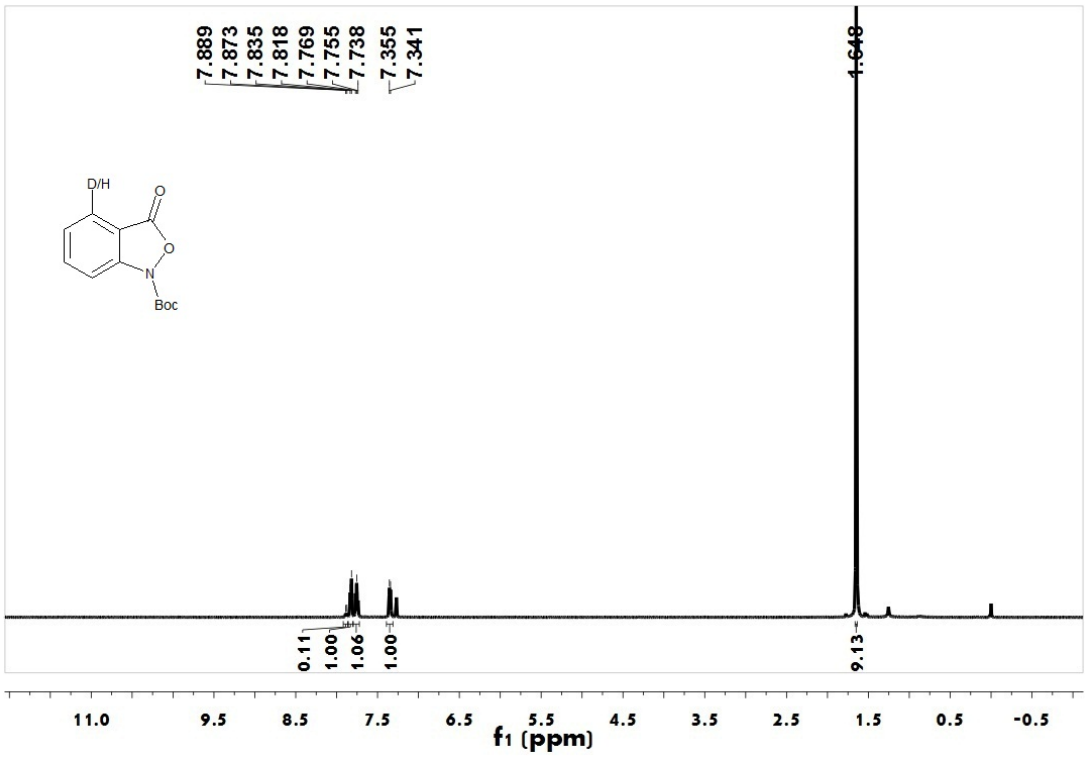
1a d_2-**1a**

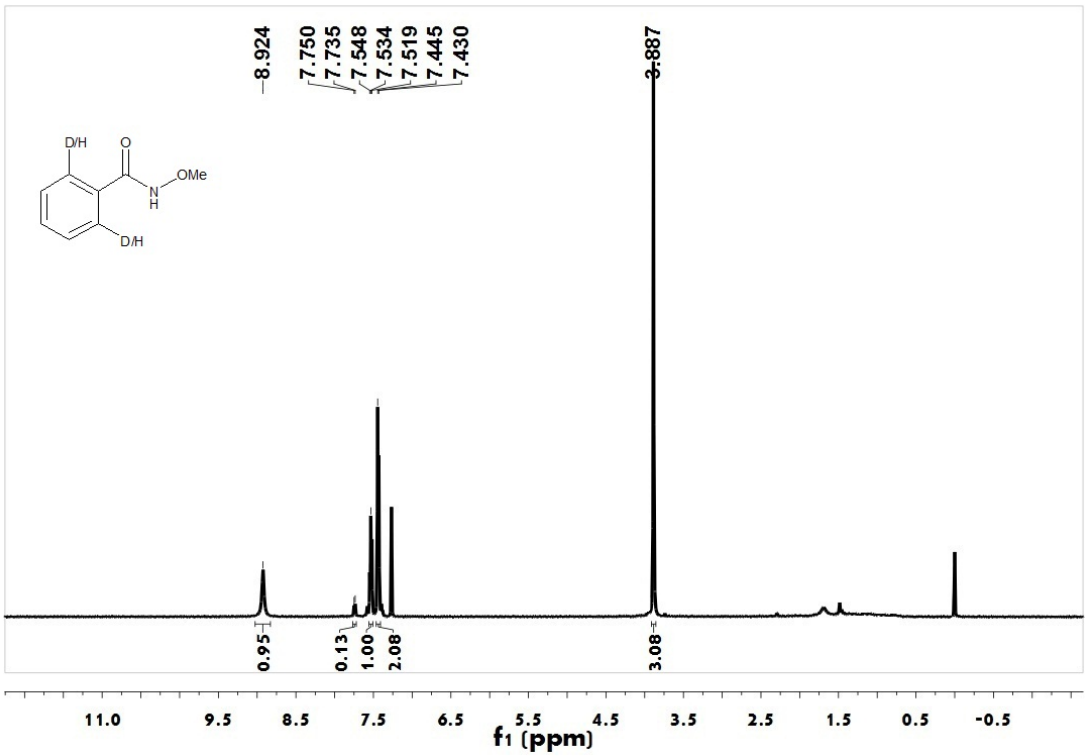
According to the general procedure, to a stirred mixture of **1a** (30.2 mg, 0.2 mmol) in CD_3OD-D_4 (1.0 mL)/actone-D_6 (1.0 mL), $[Cp^*RhCl_2]_2$ (3.1 mg, 0.005 mmol), CsOAc (11.5 mg, 0.06 mmol), CuCl (2.0 mg, 0.02 mmol) and PivOH (20.4 mg, 0.2 mmol) were added successively, the reaction mixture was stirred at room temperature for 48 h. The reaction mixture was diluted with brine (10 mL) and extracted with CH_2Cl_2 (2 × 10 mL). The combined organics were dried (Na_2SO_4), and concentrated *in vacuo*. The residue was purified by column chromatography (petroleum ether/EtOAc = 10/1, v/v) to afford the desired product d_2-**1a** yield 94% and recycle the starting material **1a** yield 6%.



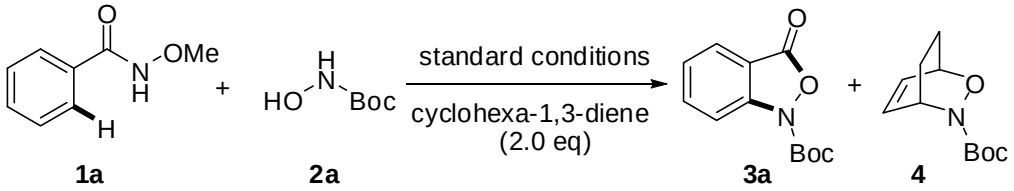


According to the general procedure, to a stirred mixture of **1a** (30.2 mg, 0.2 mmol) and **2a** (26.6 mg, 0.2 mmol) in CD_3OD-D_4 (1.0 mL)/actone-D_6 (1.0 mL), $[Cp^*RhCl_2]_2$ (3.1 mg, 0.005 mmol), CsOAc (11.5 mg, 0.06 mmol), CuCl (2.0 mg, 0.02 mmol) and PivOH (20.4 mg, 0.2 mmol) were added successively, the reaction mixture was stirred at room temperature for 15 h. The reaction mixture was diluted with brine (10 mL) and extracted with CH_2Cl_2 (2 × 10 mL). The combined organics were dried (Na_2SO_4), and concentrated *in vacuo*. The residue was purified by column chromatography (petroleum ether/EtOAc = 30/1, v/v) to afford the desired product **3a** and d-**3a** yield 15% and recycle the starting material **1a** and d_2-**1a** yield 80%. d-**3a**/**3a**=89%:11%; d_2-**1a**/**1a**=94%:6%.

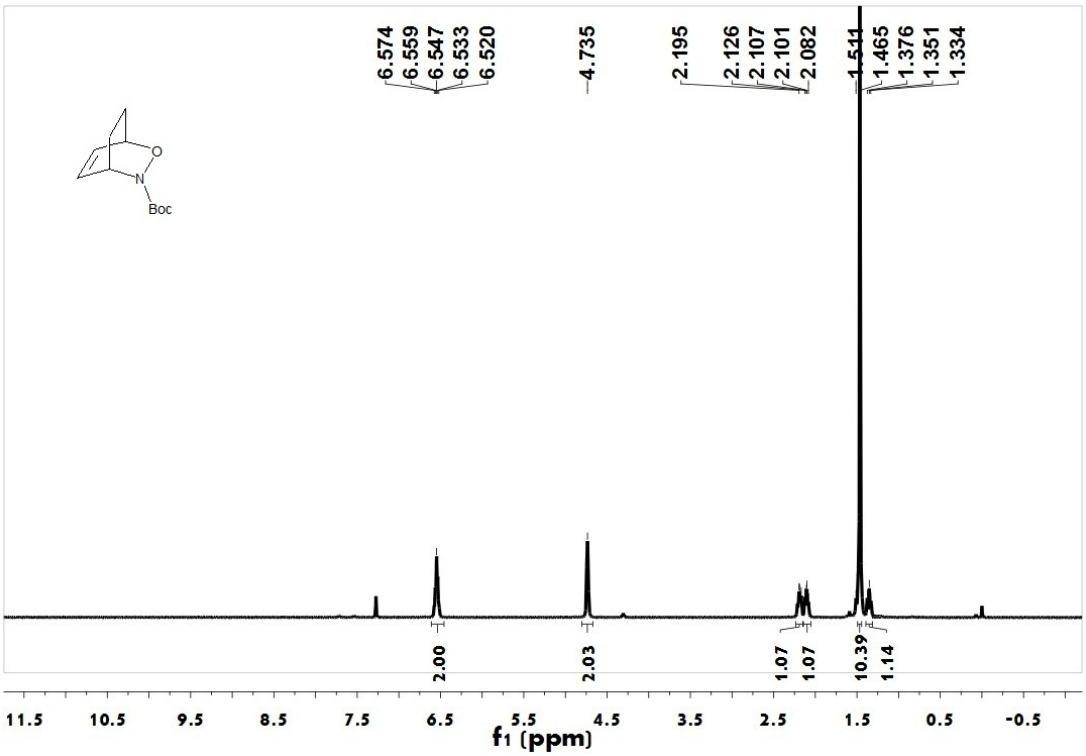


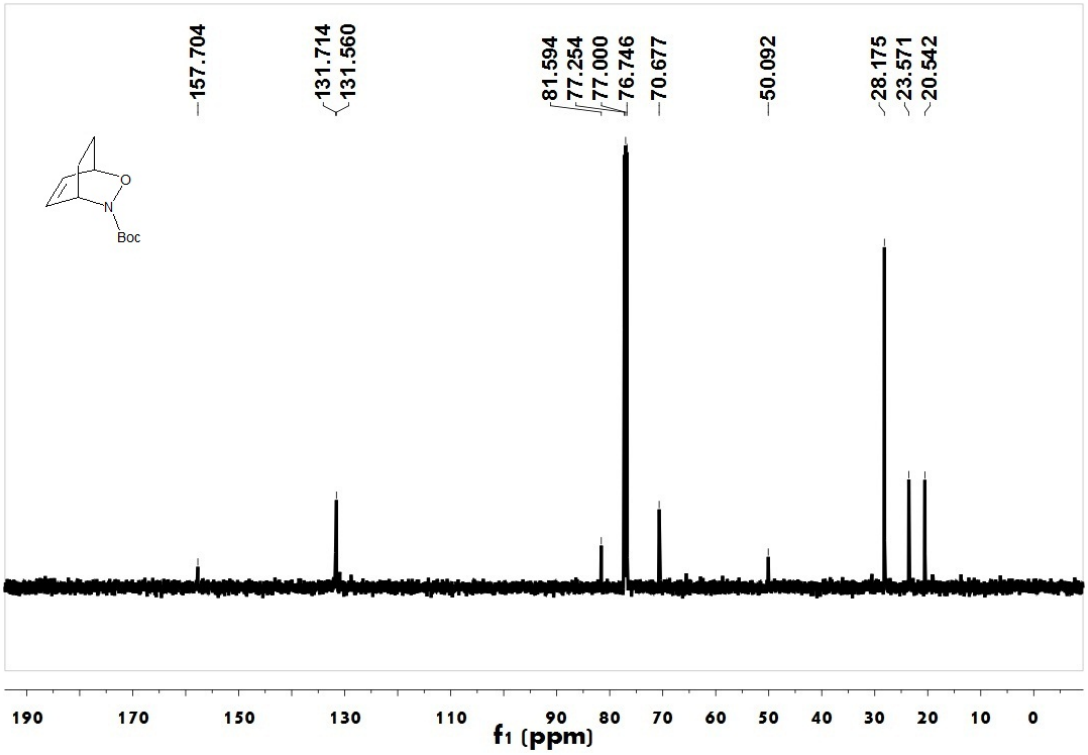


Trapping the nitrosocarbonyl compound:



According to the general procedure, to a stirred mixture of **1a** (30.2 mg, 0.2 mmol) and **2a** (31.9 mg, 0.24 mmol) in EtOH (1.0 mL)/acetone (1.0 mL), $[Cp^*RhCl_2]_2$ (3.1 mg, 0.005 mmol), CsOAc (11.5 mg, 0.06 mmol), CuCl (2.0 mg, 0.02 mmol) and PivOH (20.4 mg, 0.2 mmol) were added successively. Cyclohexa-1,3-diene (38 uL, 0.4 mmol) was added to the mixture reaction for 48 h. The reaction mixture was diluted with brine (10 mL) and extracted with CH_2Cl_2 (2 × 10 mL). The combined organics were dried (Na_2SO_4), and concentrated *in vacuo*. The residue was purified by column chromatography (petroleum ether/EtOAc = 30/1, v/v) to afford the desired product **4** yield 44% and **3a** yield 10% and recycle the starting material **1a** yield 80%.

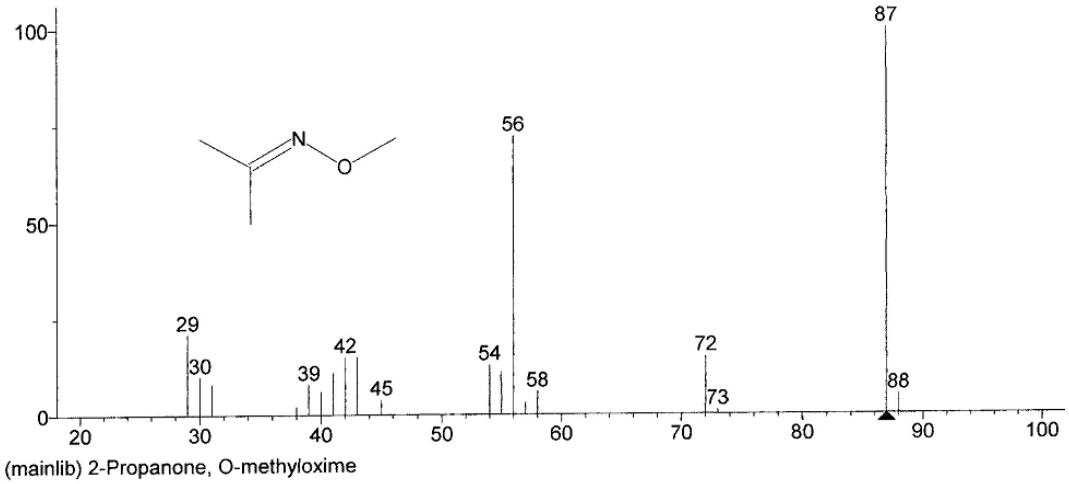

6.574
6.559
6.547
6.533
6.520
4.735
2.195
2.126
2.107
2.101
2.082
1.511
1.465
1.376
1.351
1.334
O
N
Boc
2.00
2.03
1.07
1.07
10.39
1.14
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


157.704
131.714
131.560
81.594
77.254
77.000
76.746
70.677
50.092
28.175
23.571
20.542
O
N
Boc
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

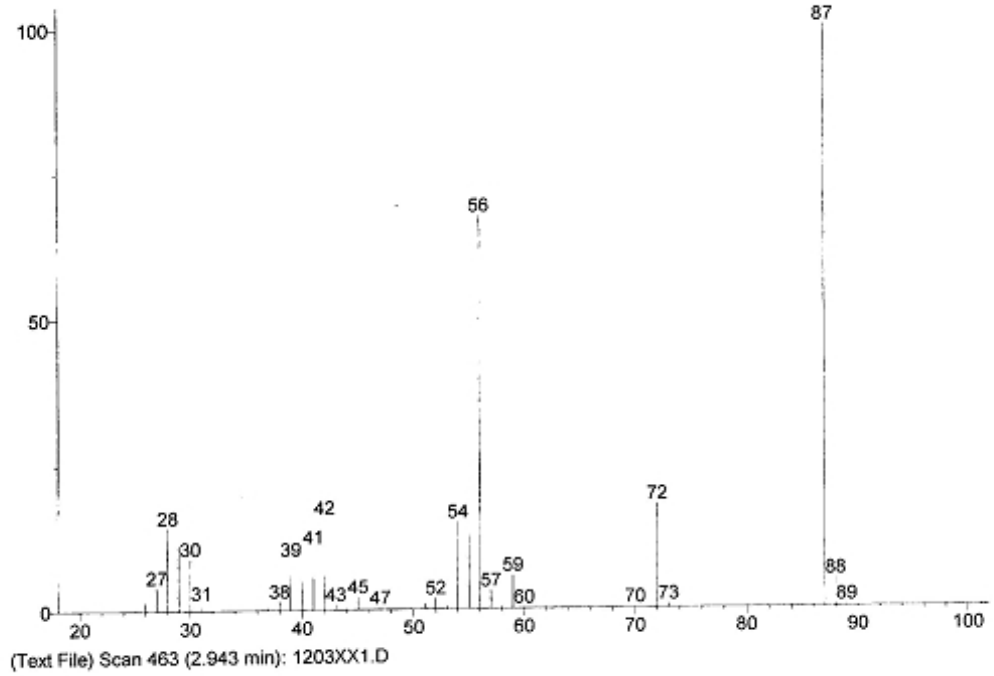
GC/MS spectrum

The reaction mixture of **3a** was filtered and then tested on a Agilent 6890/5975 spectrometer (EI).

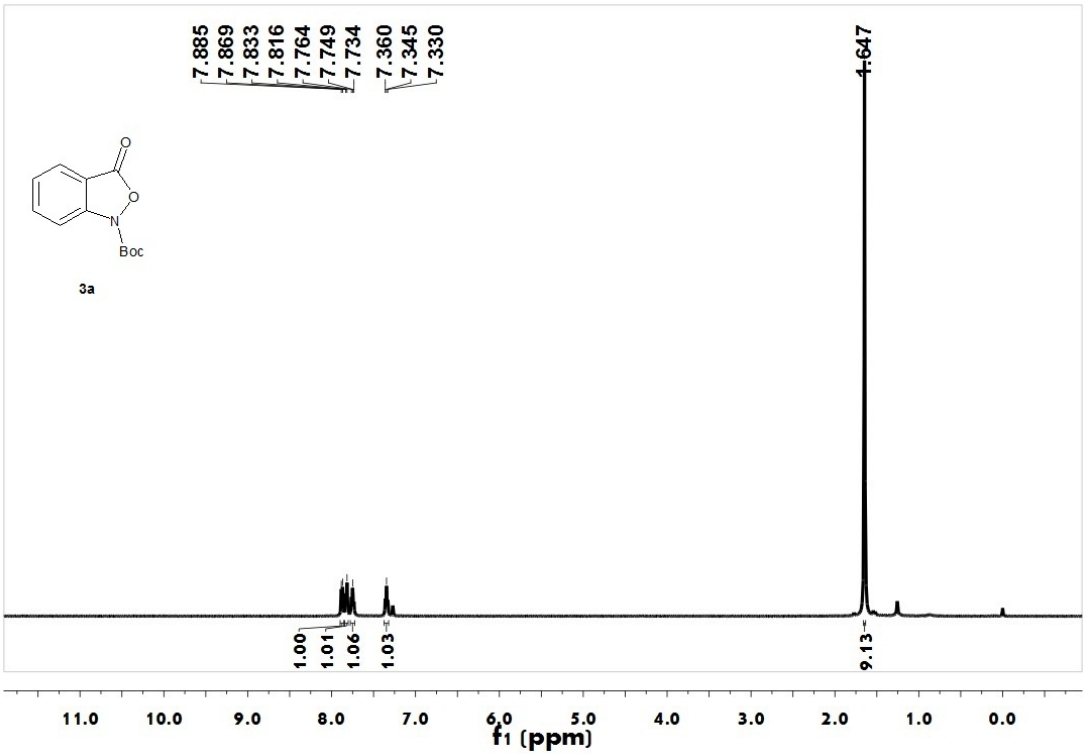
Standard sample

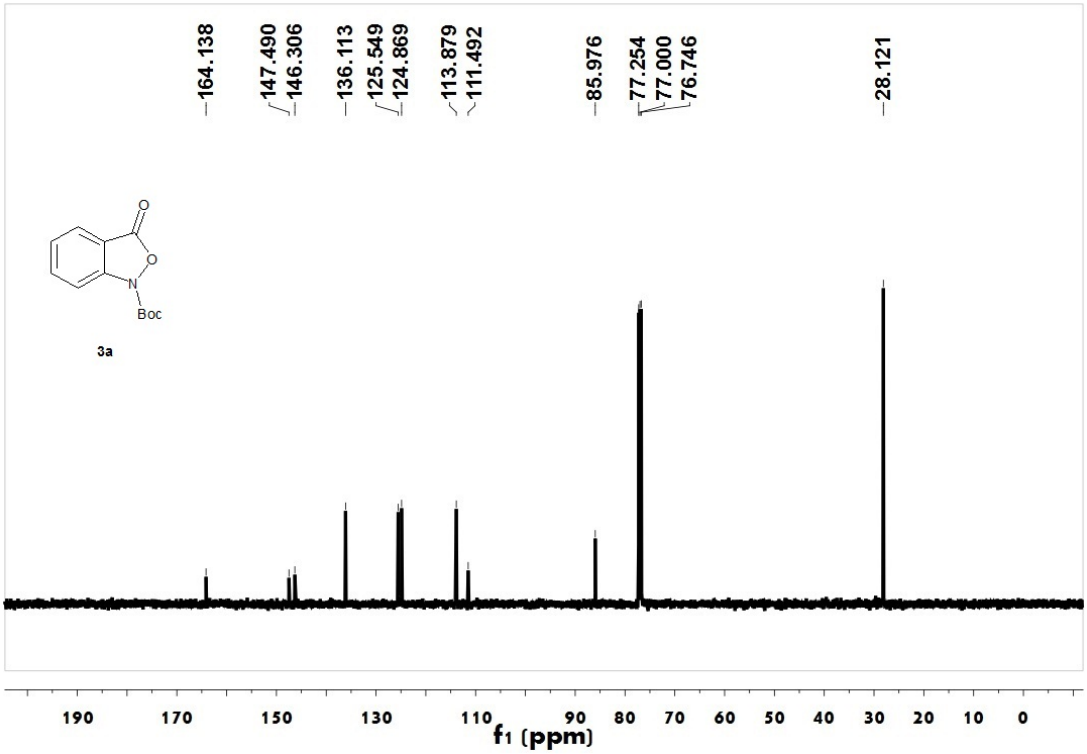


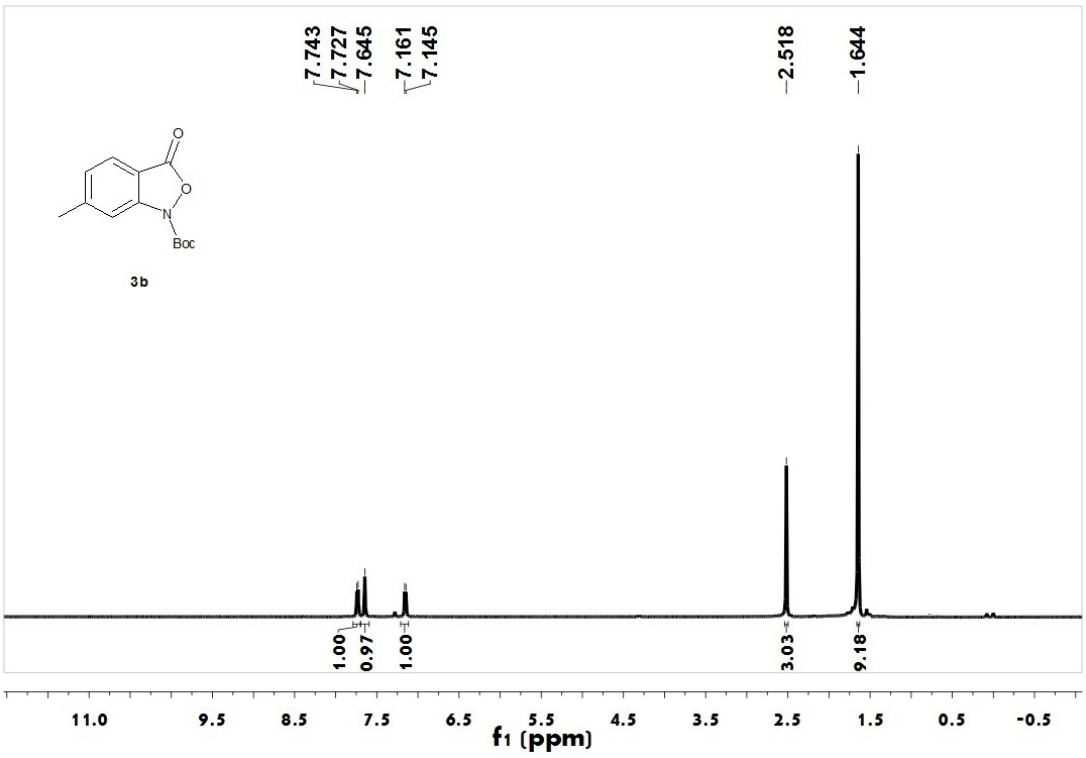
Tested sample

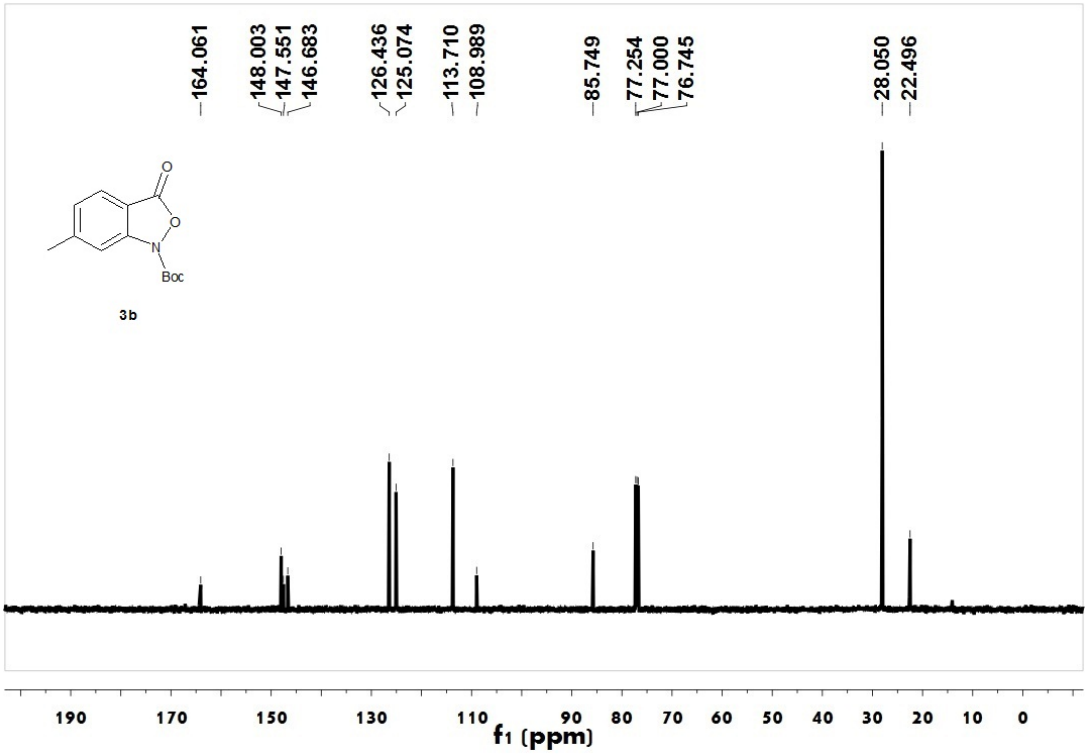


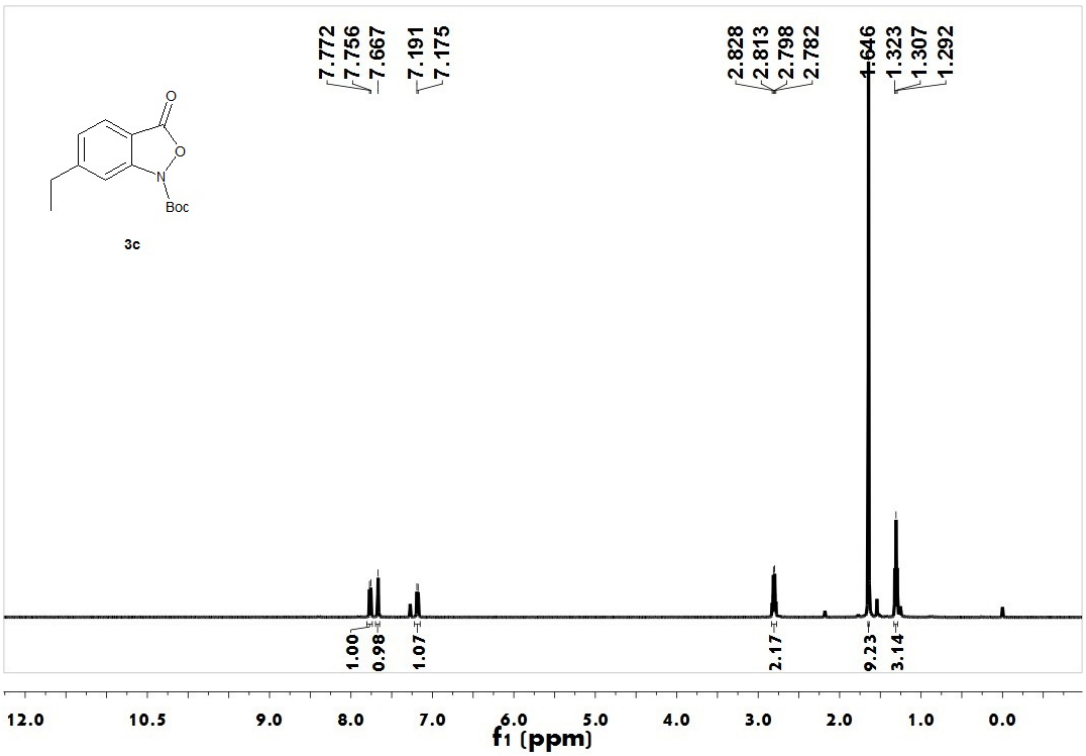
IV. Copies of ^{1}H NMR and ^{13}C NMR spectra

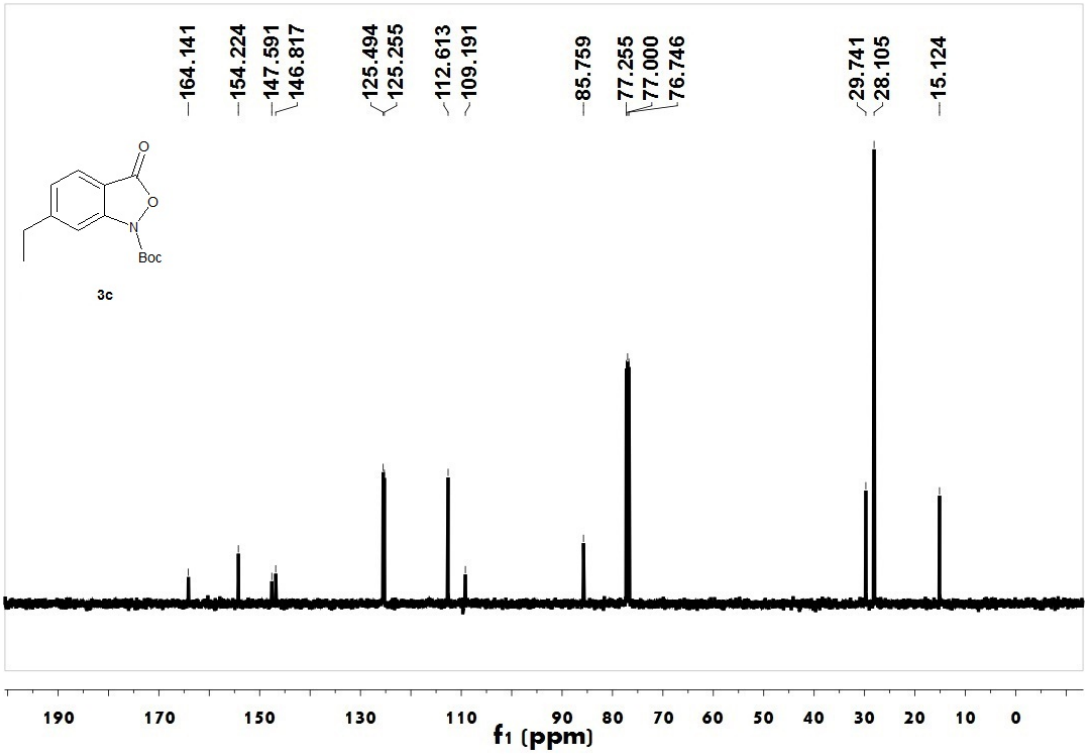


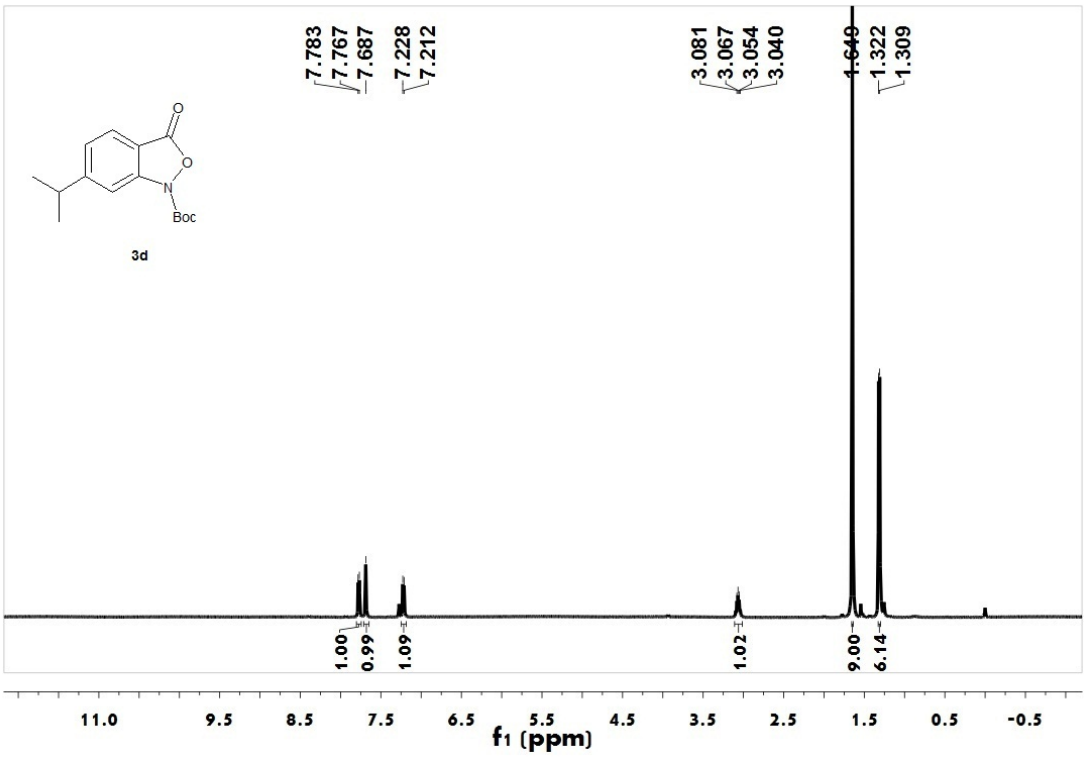


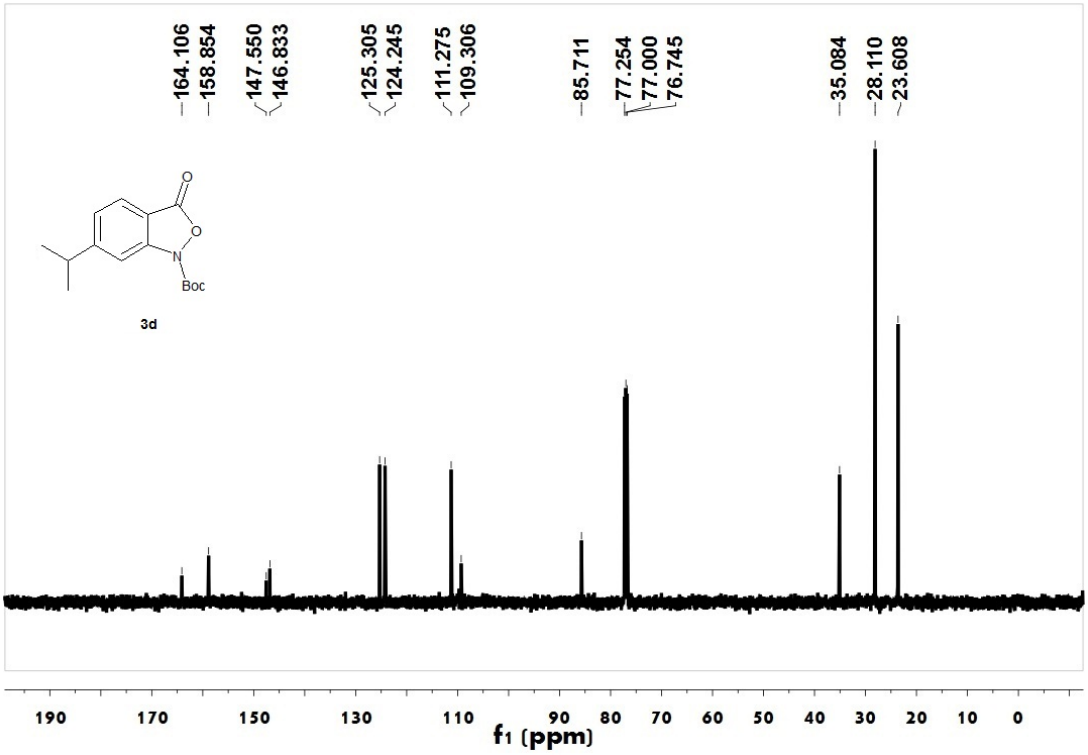

7.743
7.727
7.645
7.161
7.145
2.518
1.644
O
O
N
Boc
3b
1.00
0.97
1.00
3.03
9.18
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

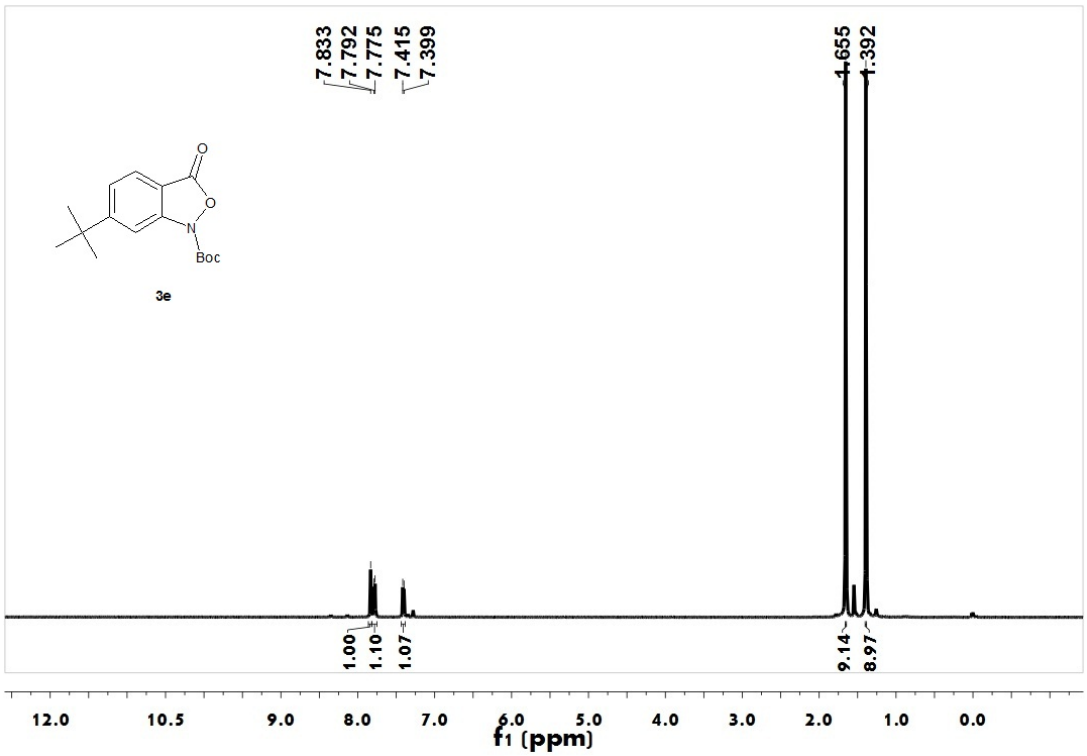

164.061
148.003
147.551
146.683
126.436
125.074
113.710
108.989
85.749
77.254
77.000
76.745
28.050
22.496
O
O
N
Boc
3b
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

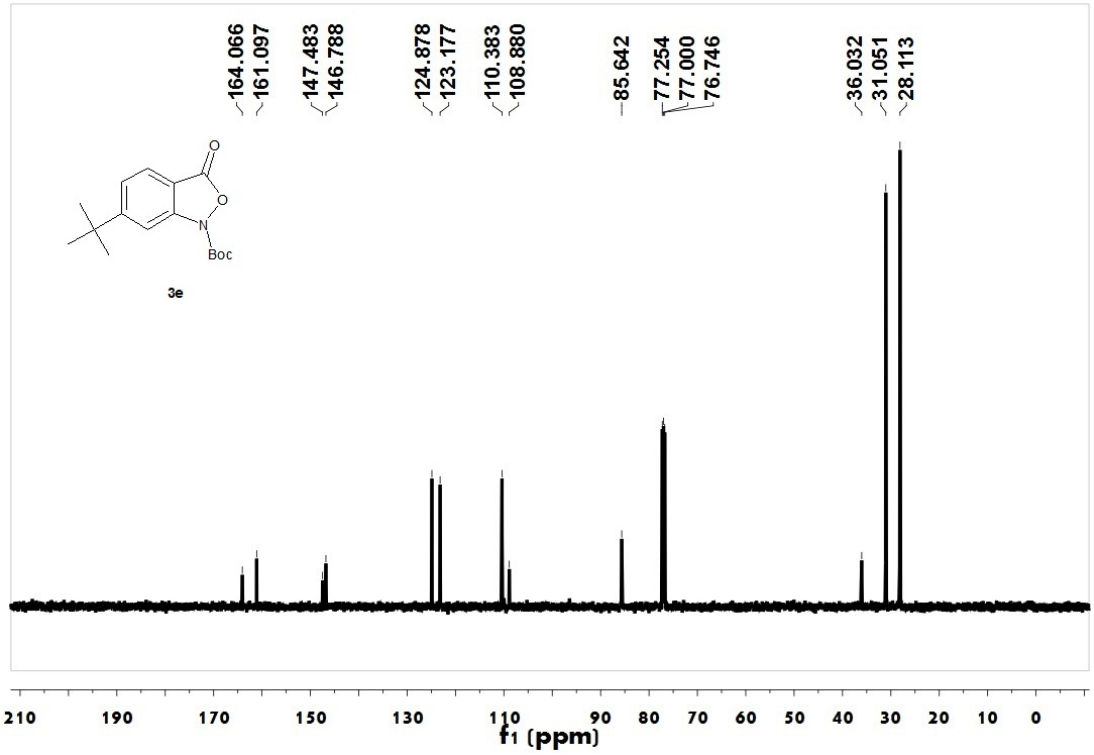

7.772
7.756
7.667
7.191
7.175
2.828
2.813
2.798
2.782
1.646
1.323
1.307
1.292
Boc
3c
1.00
0.98
1.07
2.17
9.23
3.14
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

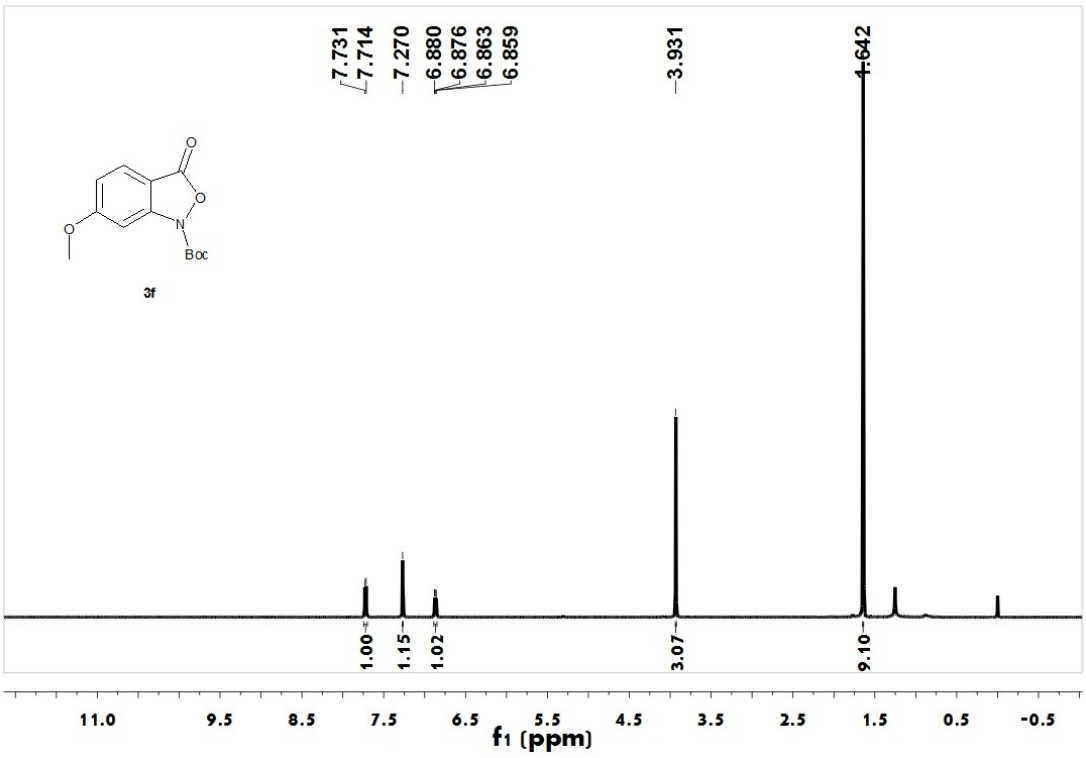

164.141
154.224
147.591
146.817
125.494
125.255
112.613
109.191
85.759
77.255
77.000
76.746
29.741
28.105
15.124
Boc
3c
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

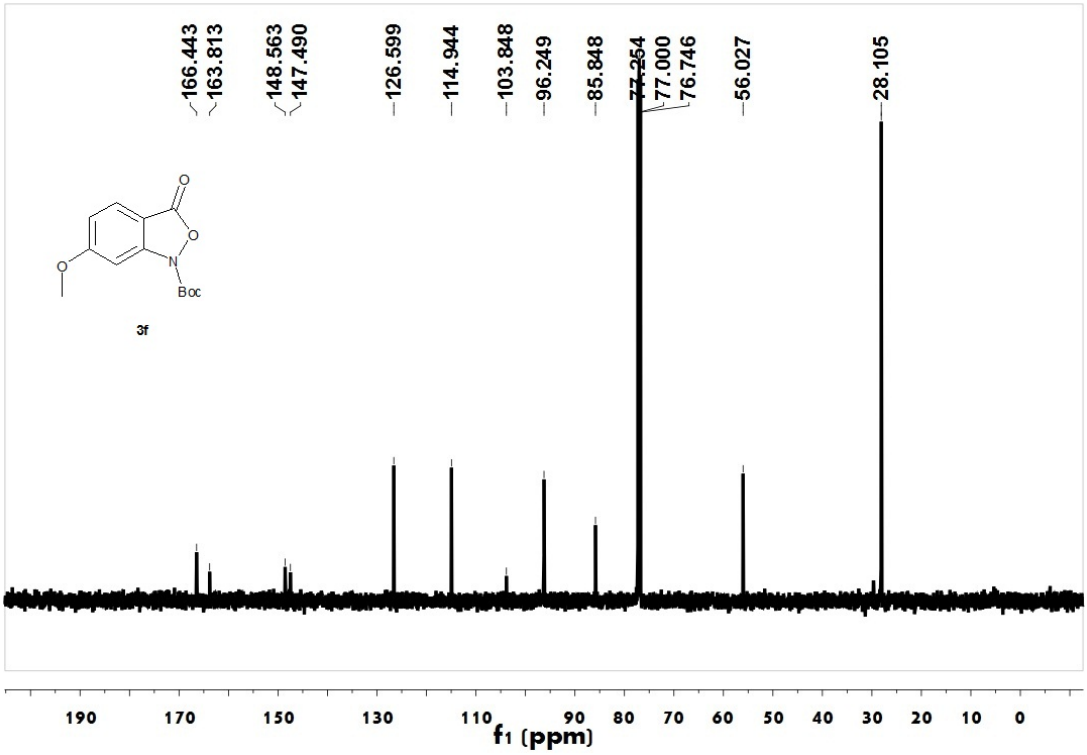

7.783
7.767
7.687
7.228
7.212
3.081
3.067
3.054
3.040
1.640
1.322
1.309
O
O
N
Boc
3d
1.00
0.99
1.09
1.02
9.00
6.14
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

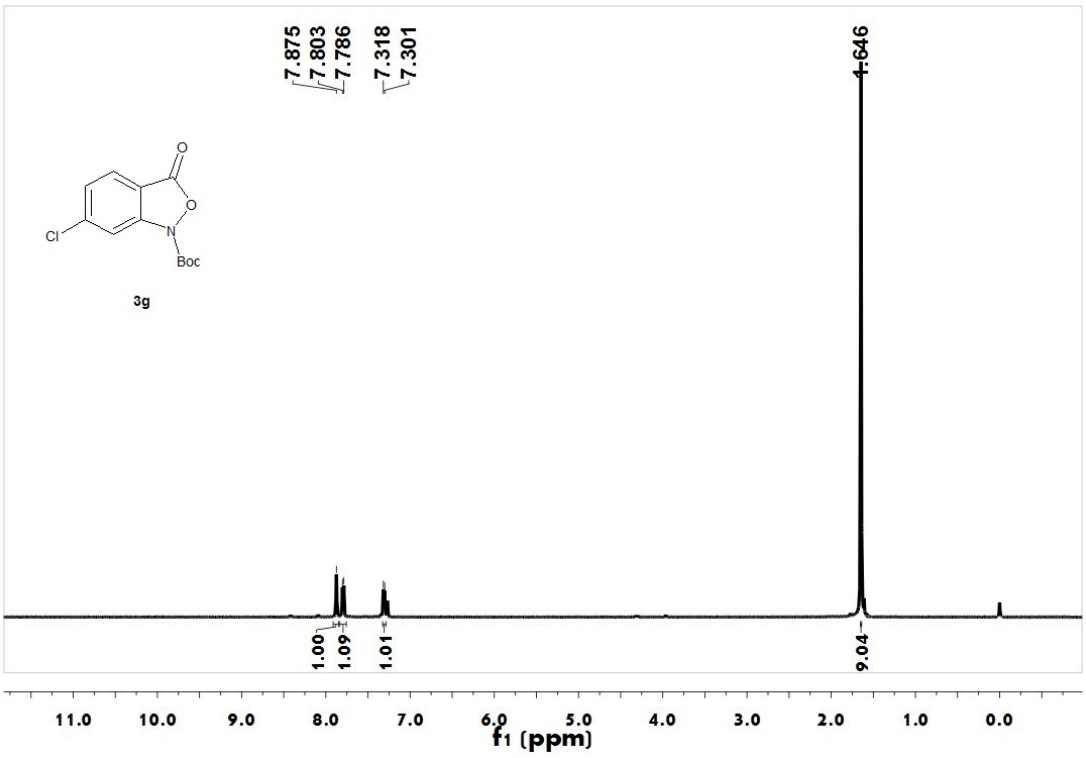

164.106
158.854
147.550
146.833
125.305
124.245
111.275
109.306
85.711
77.254
77.000
76.745
35.084
28.110
23.608
O
O
N
Boc
3d
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

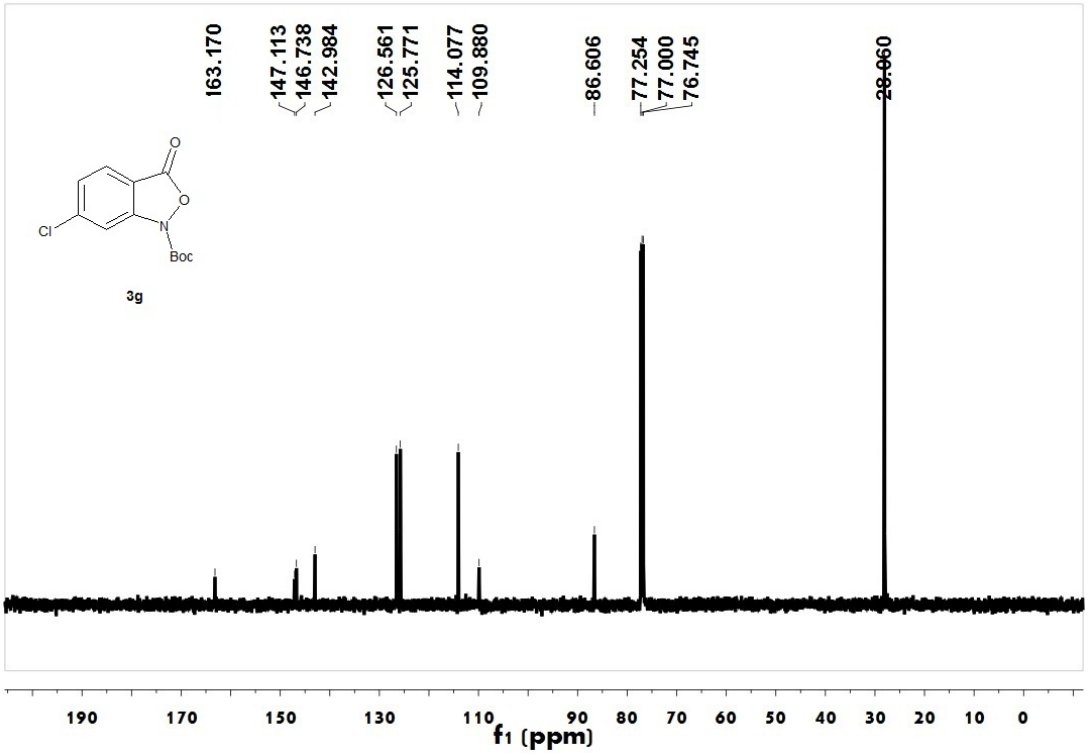

7.833
7.792
7.775
7.415
7.399
1.655
1.392
Boc
3e
1.00
1.10
1.07
9.14
8.97
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

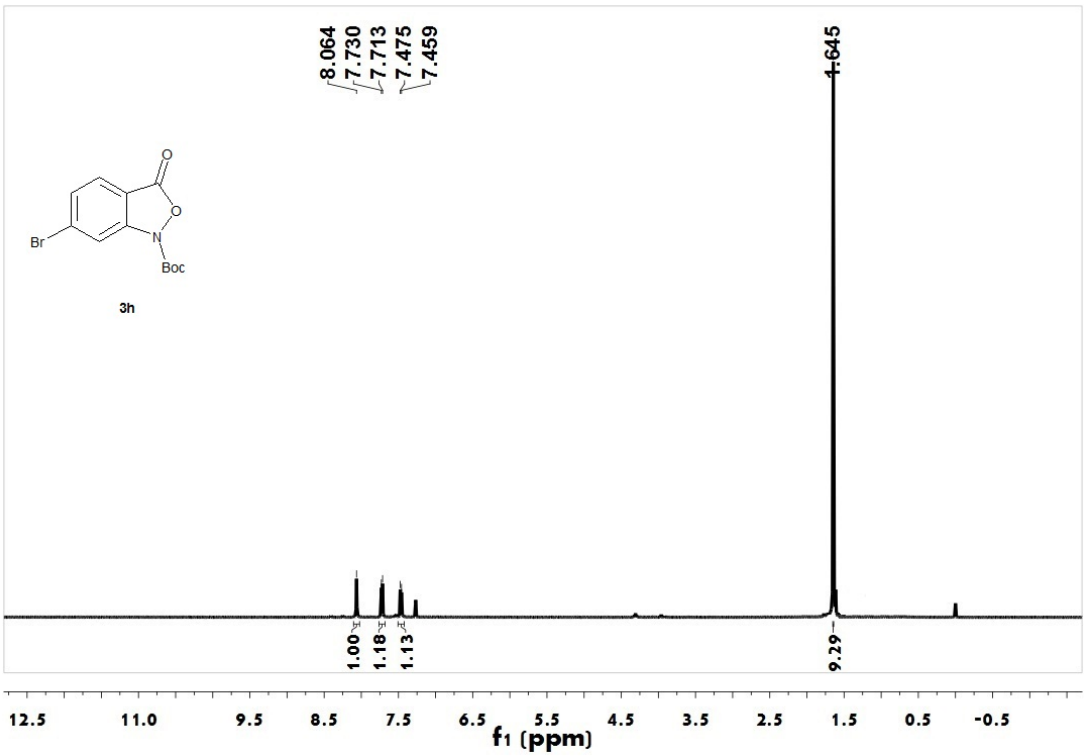

164.066
161.097
147.483
146.788
124.878
123.177
110.383
108.880
85.642
77.254
77.000
76.746
36.032
31.051
28.113
Boc
3e
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

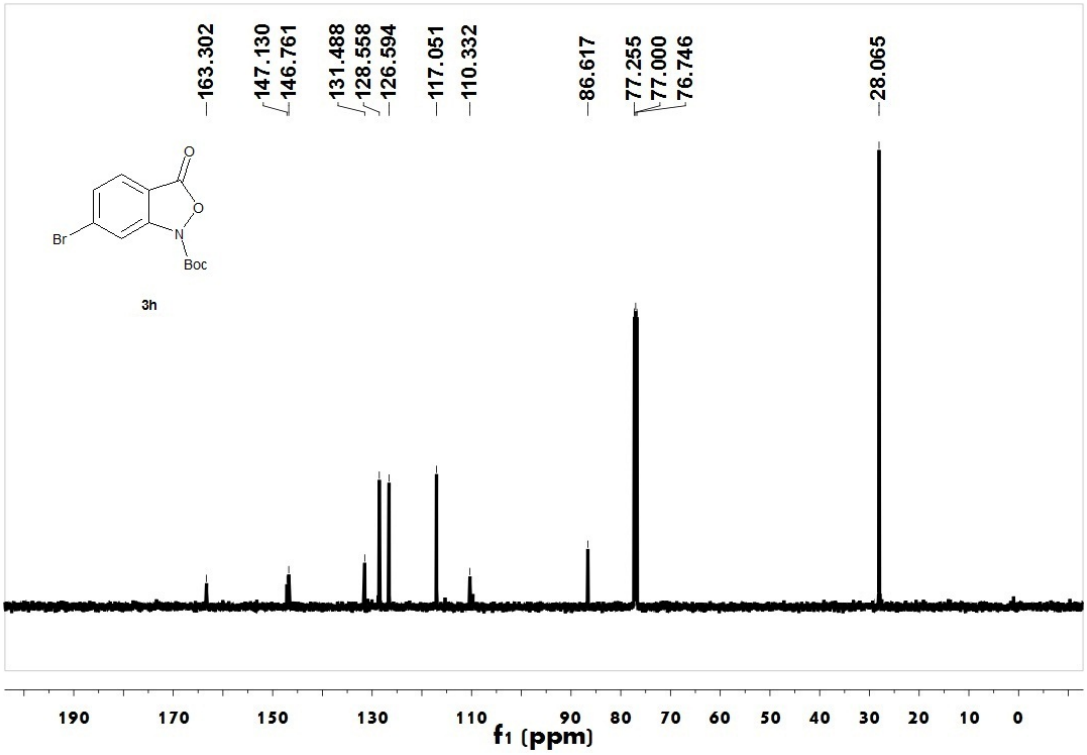

7.731
7.714
7.270
6.880
6.876
6.863
6.859
3.931
1.642
Boc
3f
1.00
1.15
1.02
3.07
9.10
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

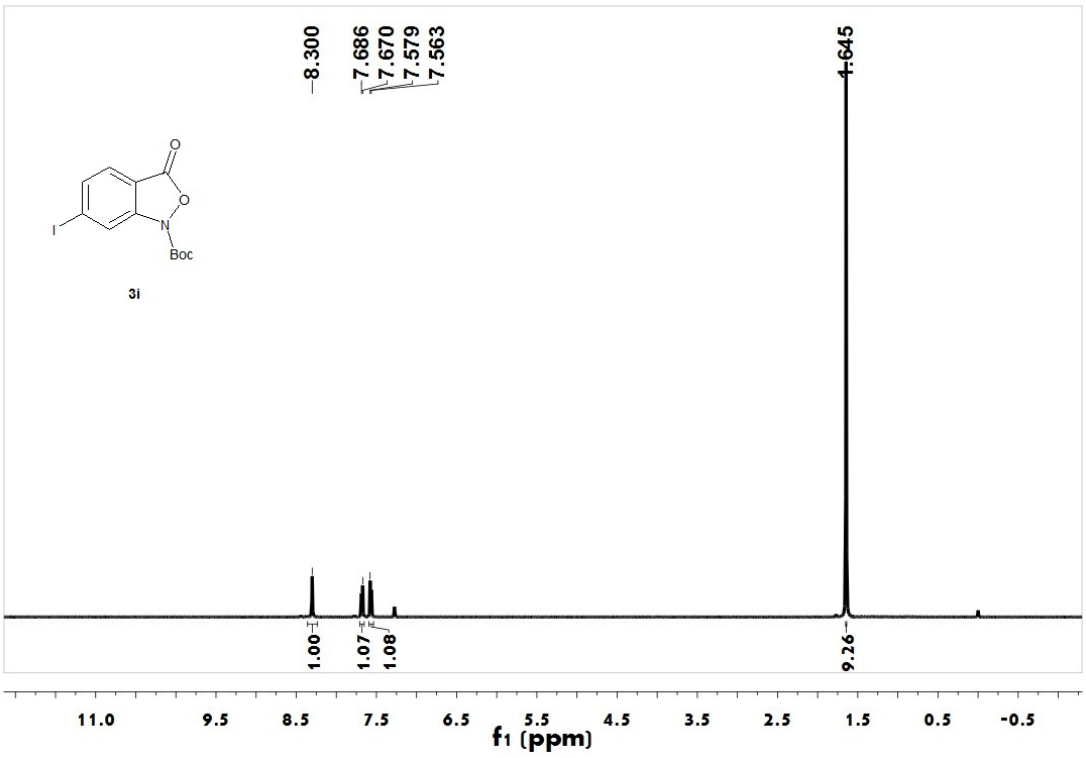

166.443
163.813
148.563
147.490
126.599
114.944
103.848
96.249
85.848
77.254
77.000
76.746
56.027
28.105
Boc
3f
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

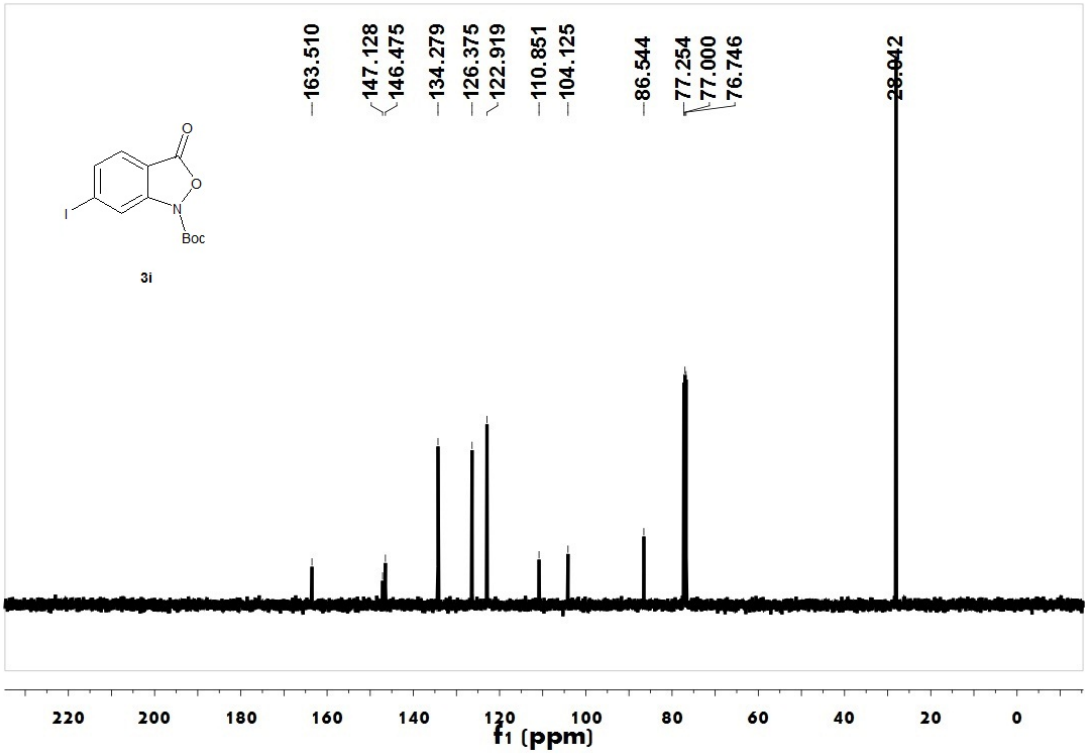


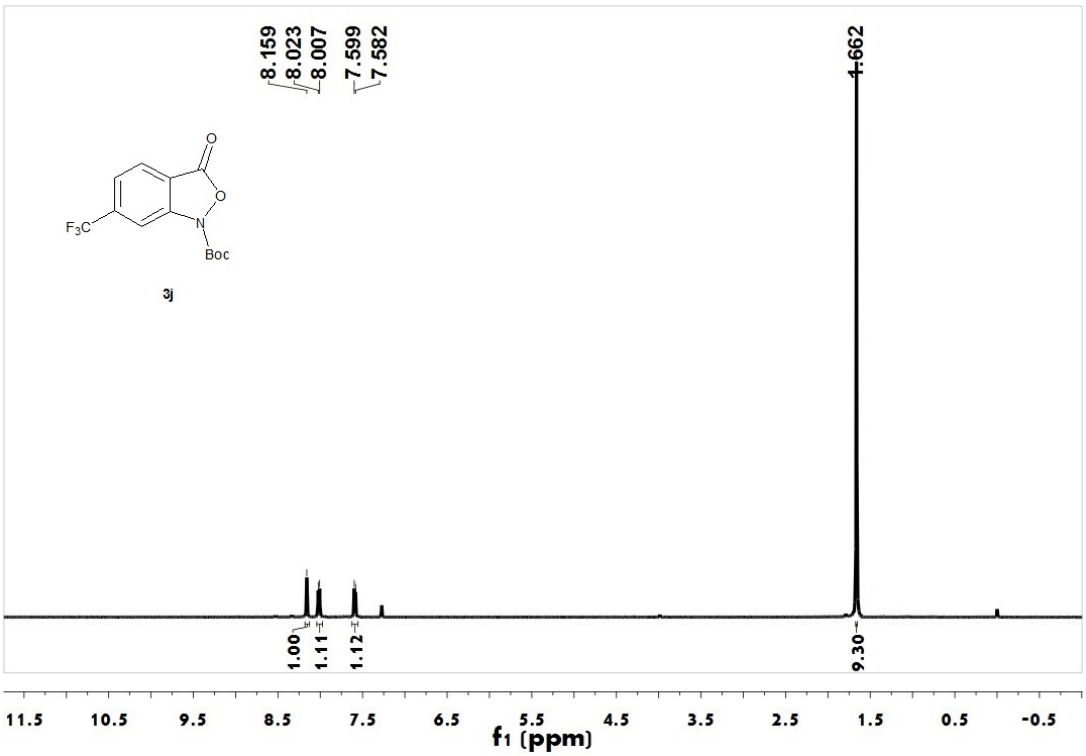


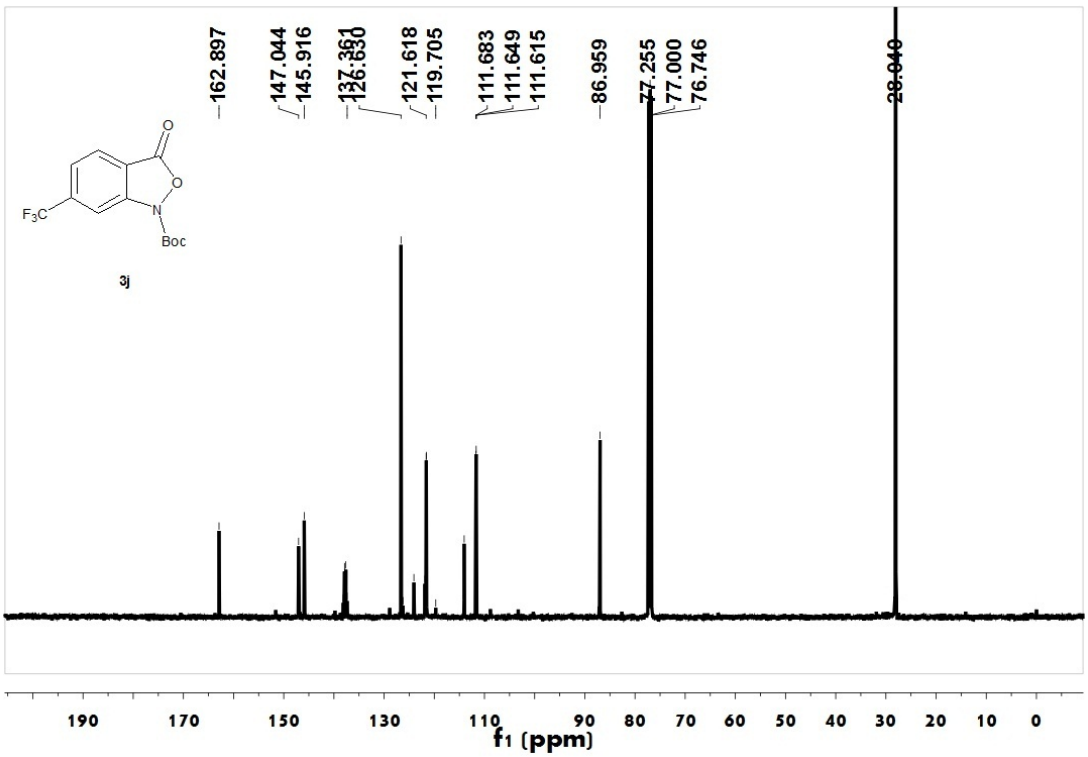

8.064
7.730
7.713
7.475
7.459
4.645
Br
O
O
N
Boc
3h
1.00
1.18
1.13
9.29
12.5
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

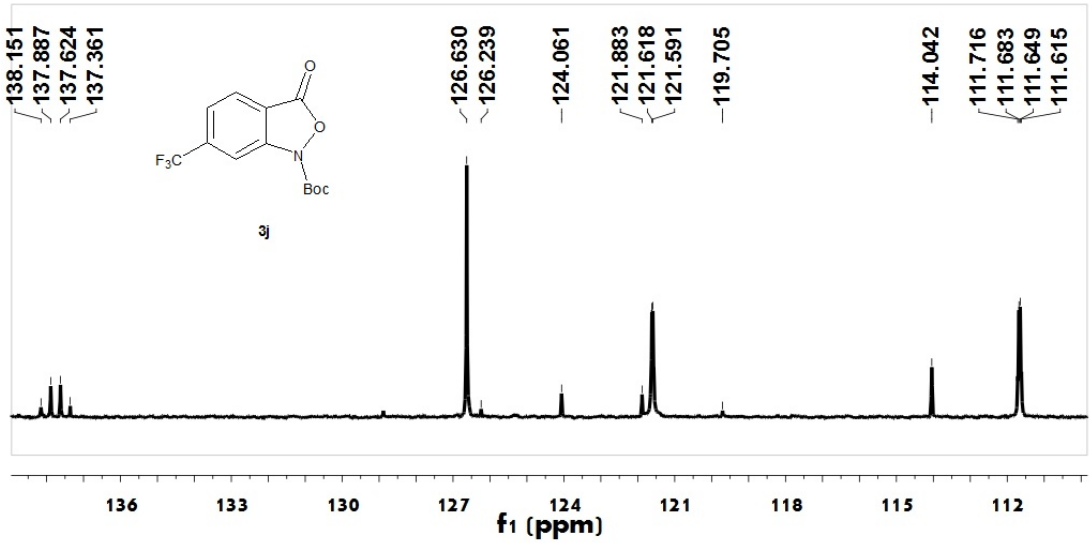

163.302
147.130
146.761
131.488
128.558
126.594
117.051
110.332
86.617
77.255
77.000
76.746
28.065
Br
O
O
N
Boc
3h
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

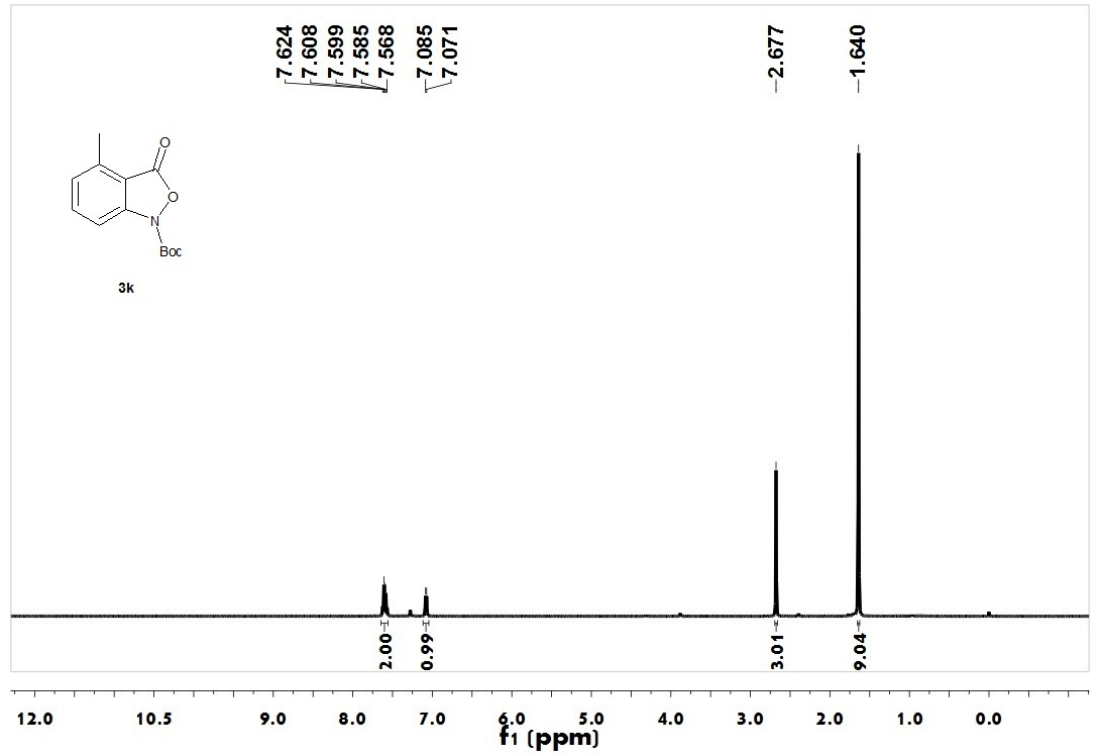

8.300
7.686
7.670
7.579
7.563
4.645
1.00
1.07
1.08
9.26
3i
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

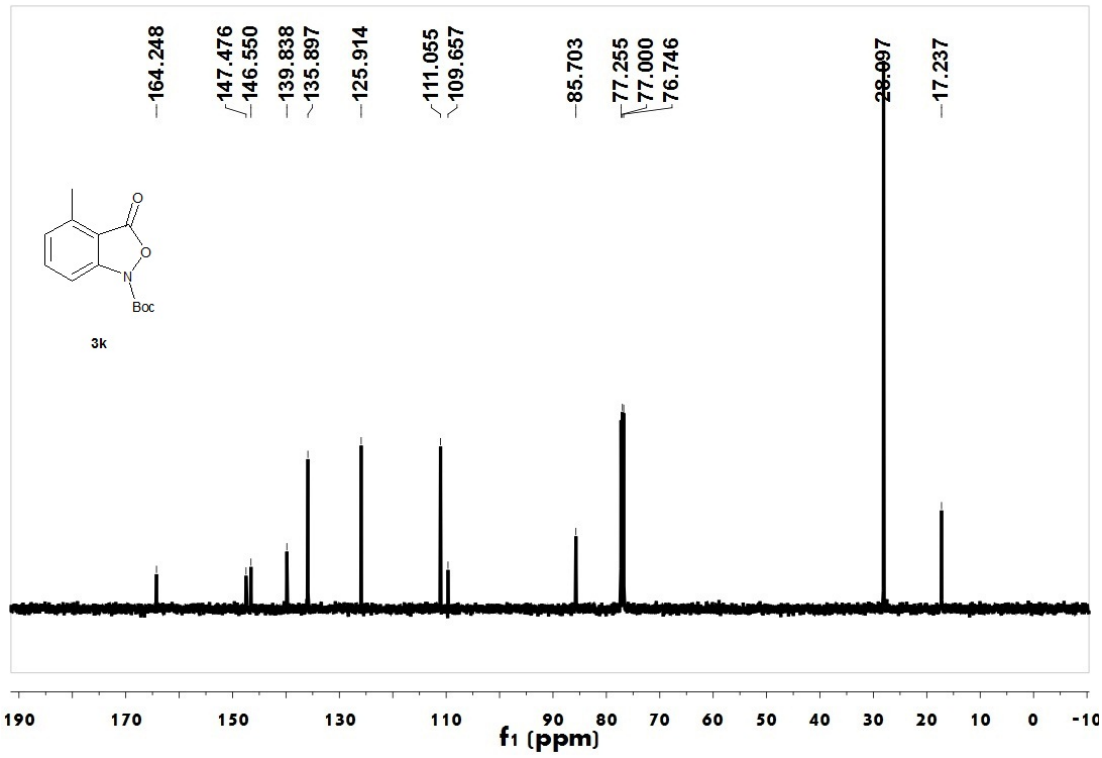

163.510
147.128
146.475
134.279
126.375
122.919
110.851
104.125
86.544
77.254
77.000
76.746
28.042
3i
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

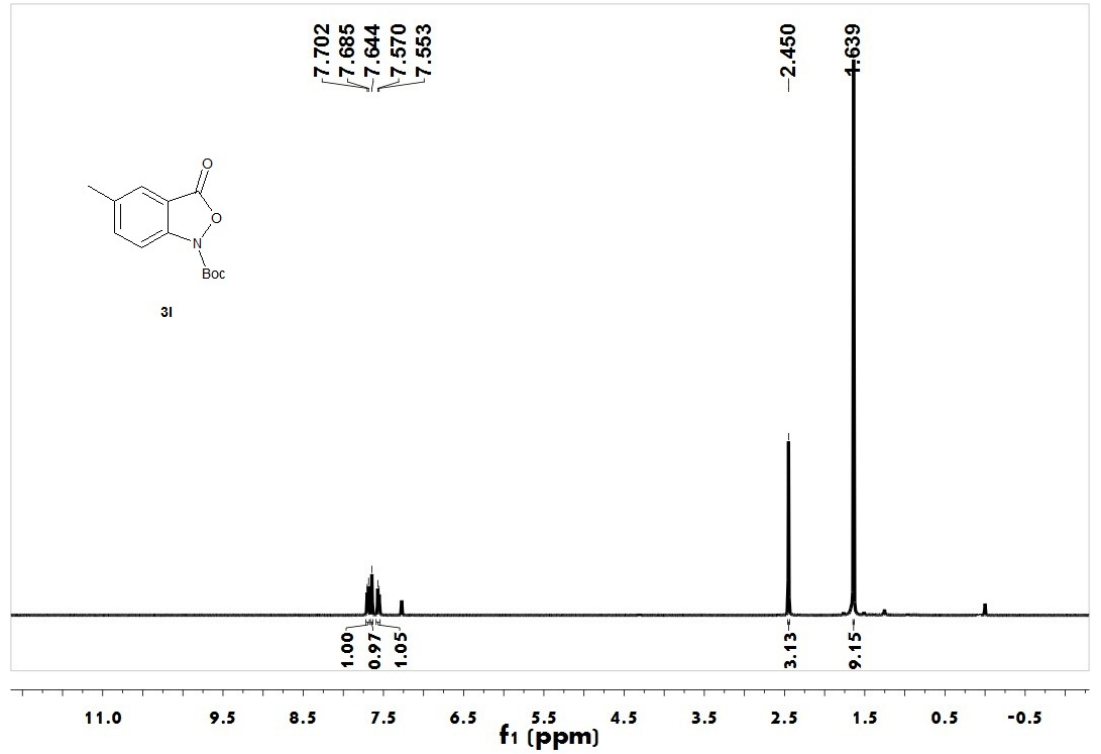

8.159
8.023
8.007
7.599
7.582
1.662
F3C
O
O
N
Boc
3j
1.00
1.11
1.12
9.30
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

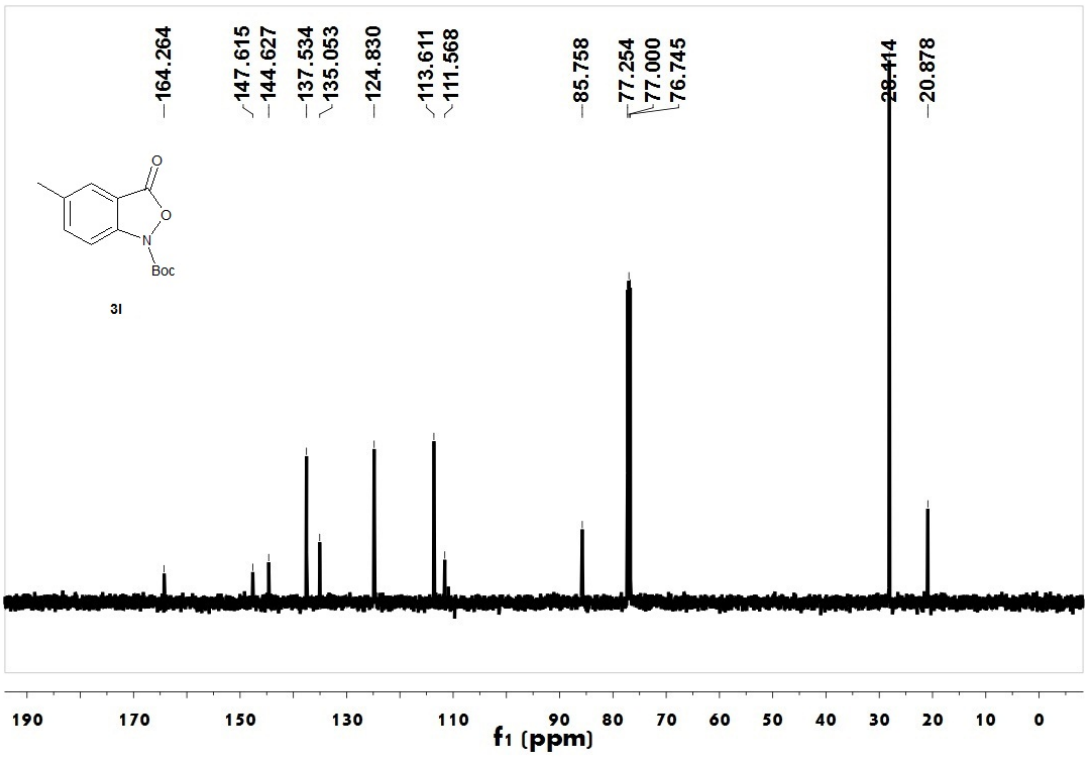

162.897
147.044
145.916
137.361
126.830
121.618
119.705
111.683
111.649
111.615
86.959
77.255
77.000
76.746
28.040
F3C
O
O
N
Boc
3j
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

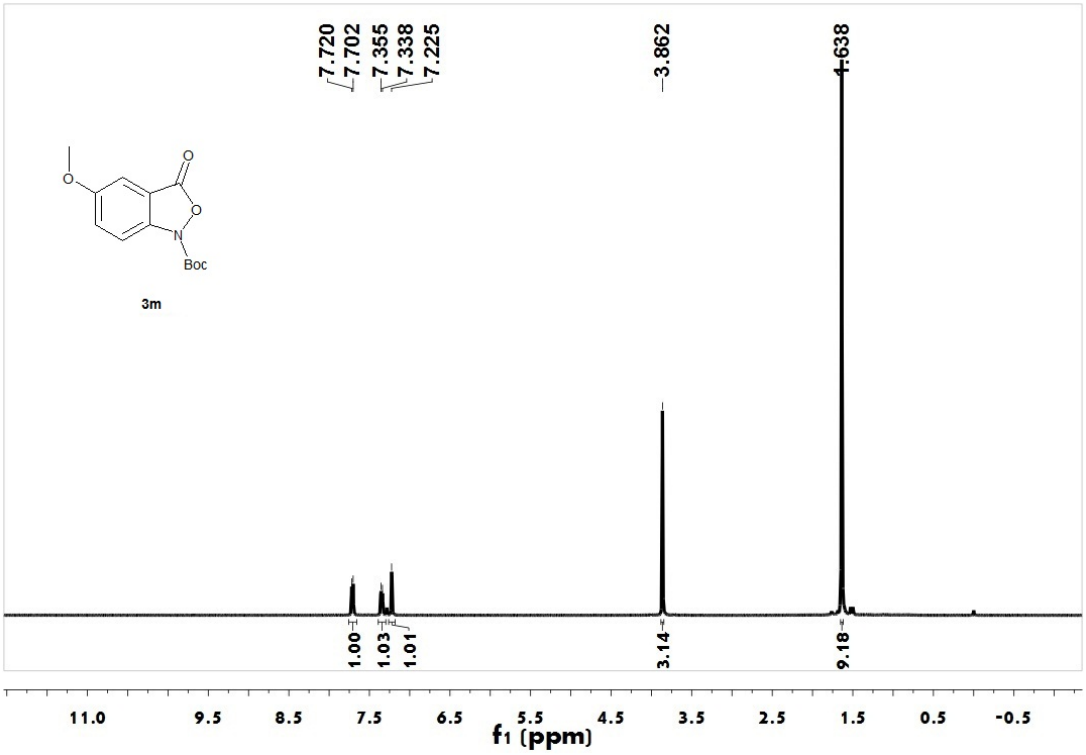

138.151
137.887
137.624
137.361
126.630
126.239
124.061
121.883
121.618
121.591
119.705
114.042
111.716
111.683
111.649
111.615
F3C
O
O
N
Boc
3j
136
133
130
127
124
121
118
115
112
f1 (ppm)

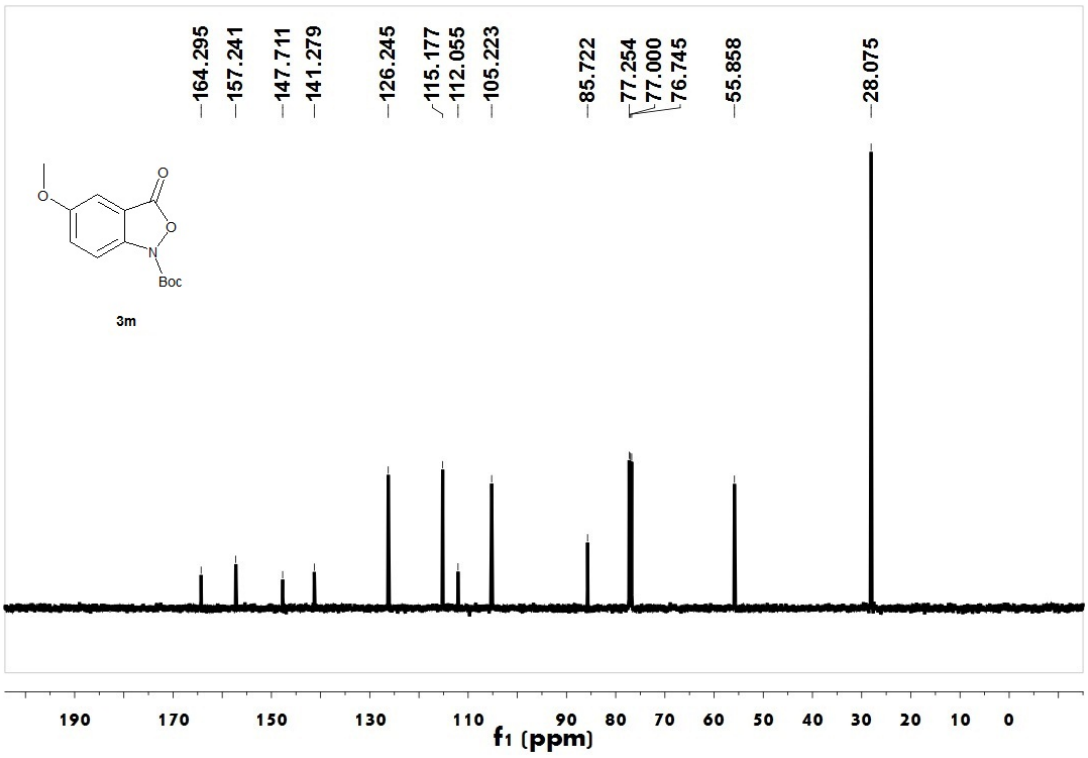

7.624
7.608
7.599
7.585
7.568
7.085
7.071
2.677
1.640
O
O
N
Boc
3k
2.00
0.99
3.01
9.04
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

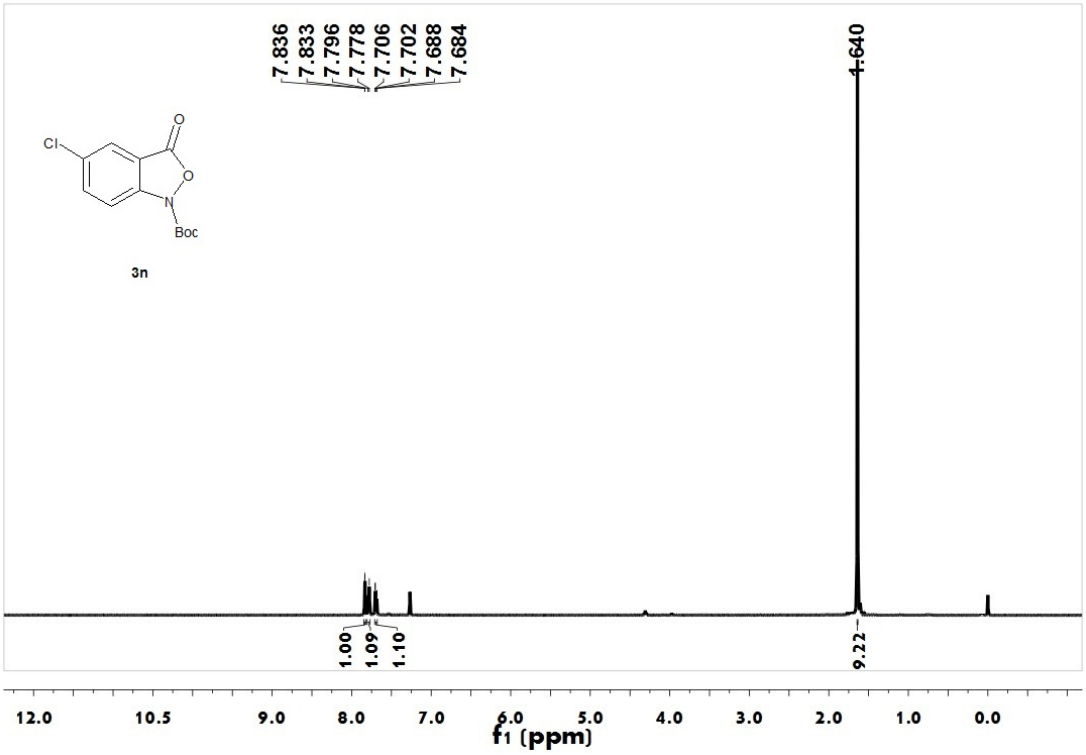

164.248
147.476
146.550
139.838
135.897
125.914
111.055
109.657
85.703
77.255
77.000
76.746
28.097
17.237
O
O
N
Boc
3k
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

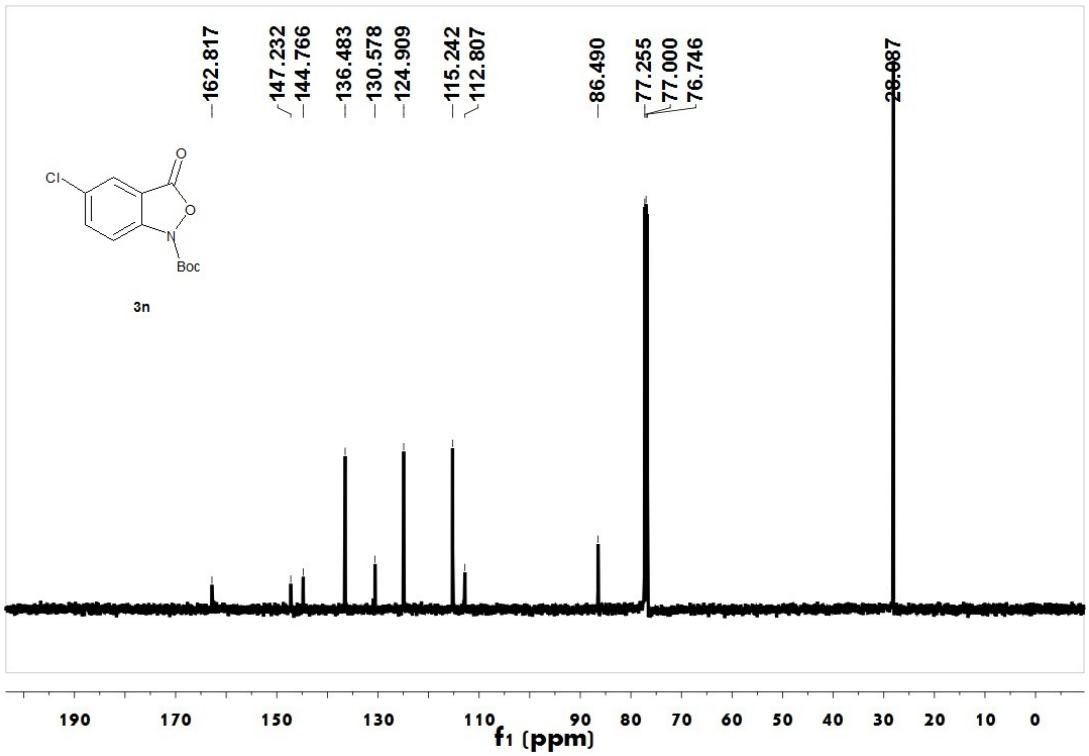

7.702
7.685
7.644
7.570
7.553
2.450
1.639
O
O
N
Boc
3l
1.00
0.97
1.05
3.13
9.15
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

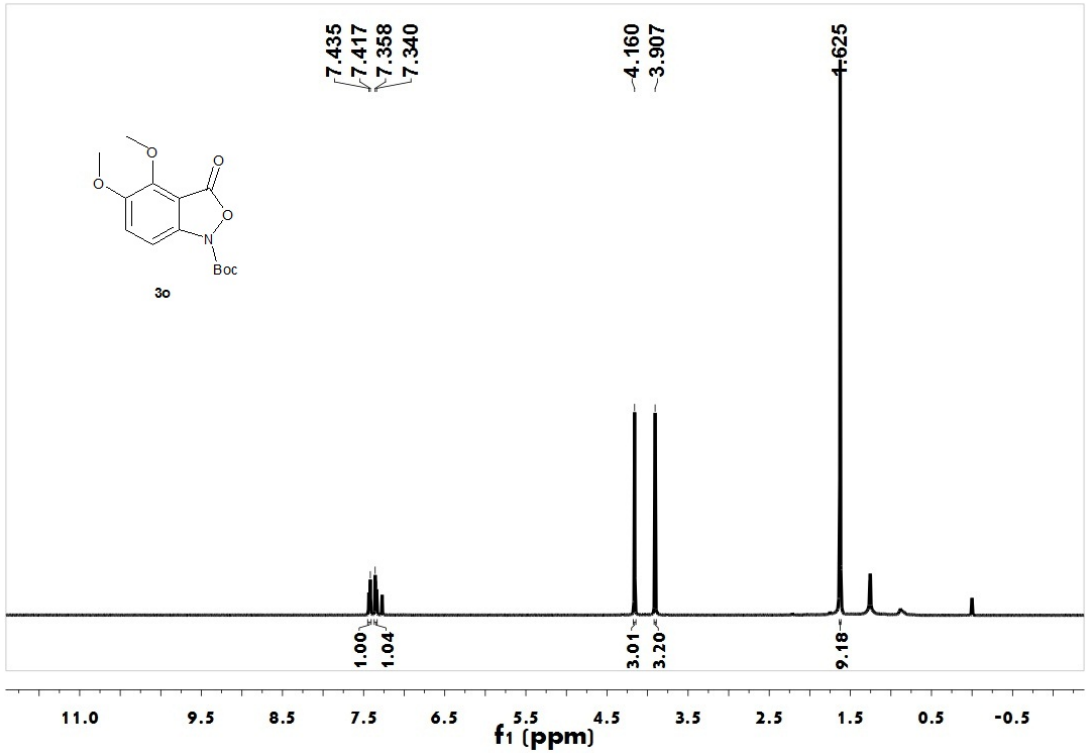


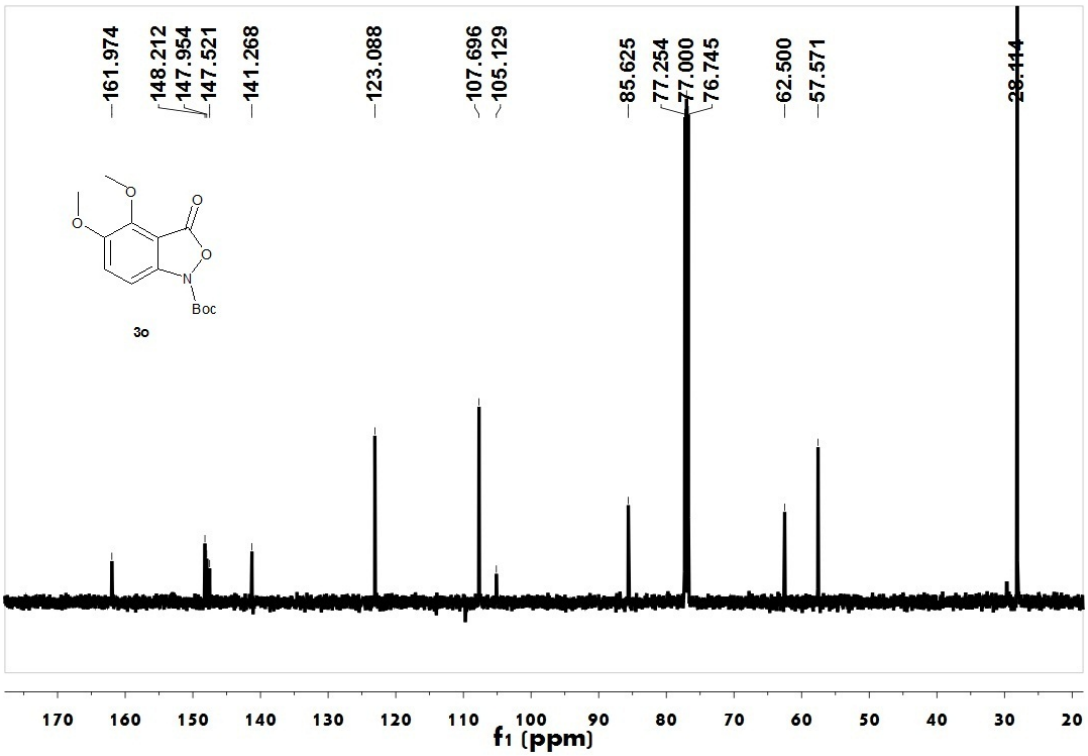


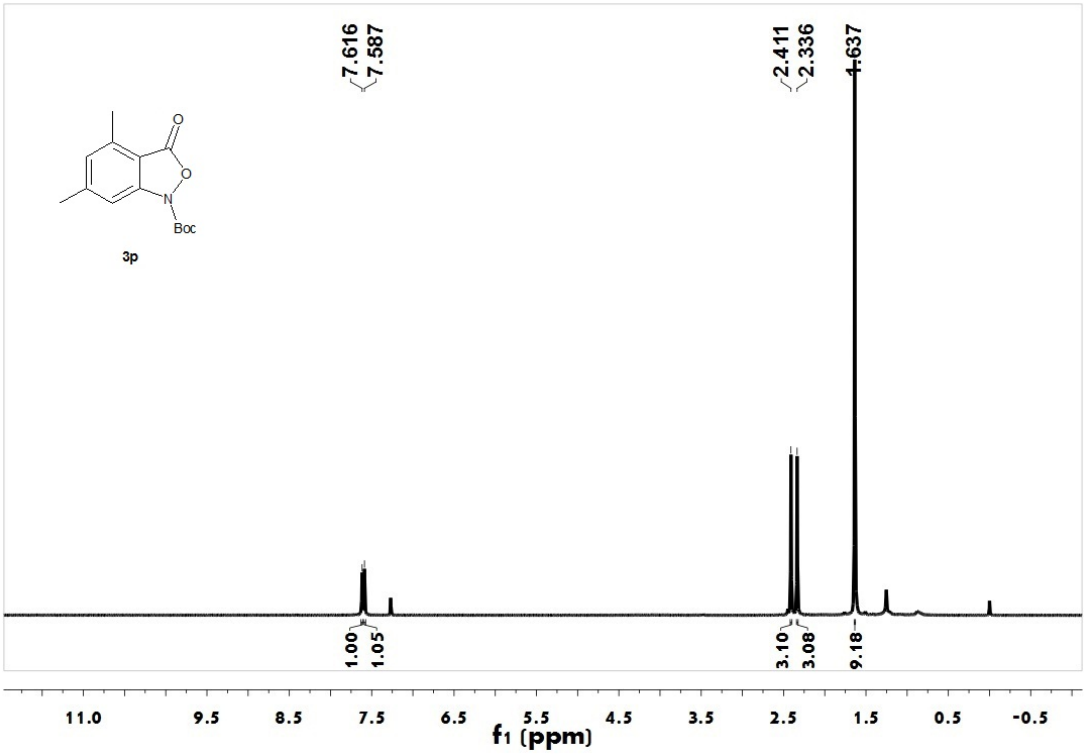

164.295
157.241
147.711
141.279
126.245
115.177
112.055
105.223
85.722
77.254
77.000
76.745
55.858
28.075
O
O
O
N
Boc
3m
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

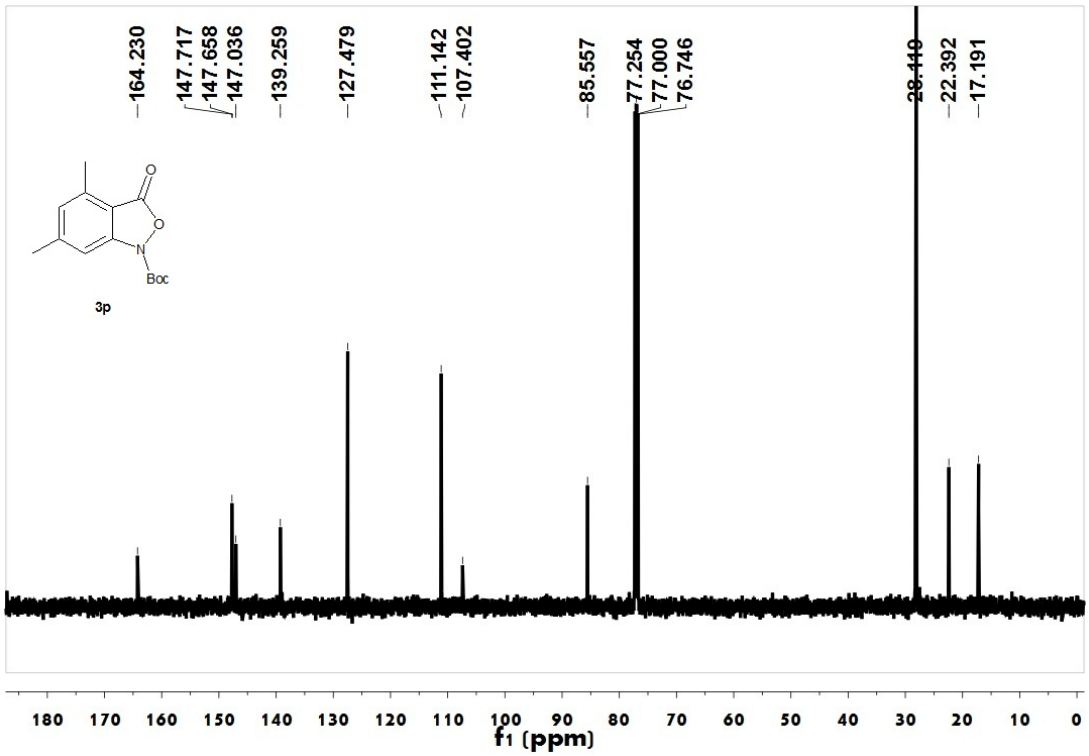

7.836
7.833
7.796
7.778
7.706
7.702
7.688
7.684
1.640
Cl
O
O
N
Boc
3n
1.00
1.09
1.10
9.22
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

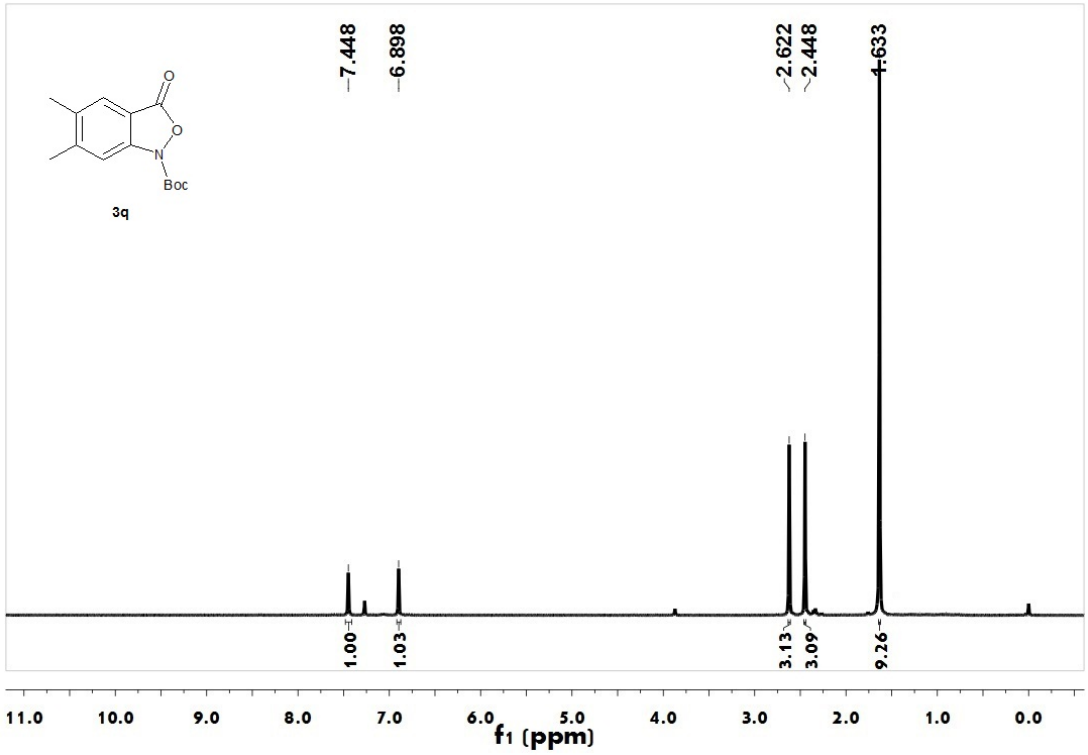

162.817
147.232
144.766
136.483
130.578
124.909
115.242
112.807
86.490
77.255
77.000
76.746
28.087
Cl
O
O
N
Boc
3n
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

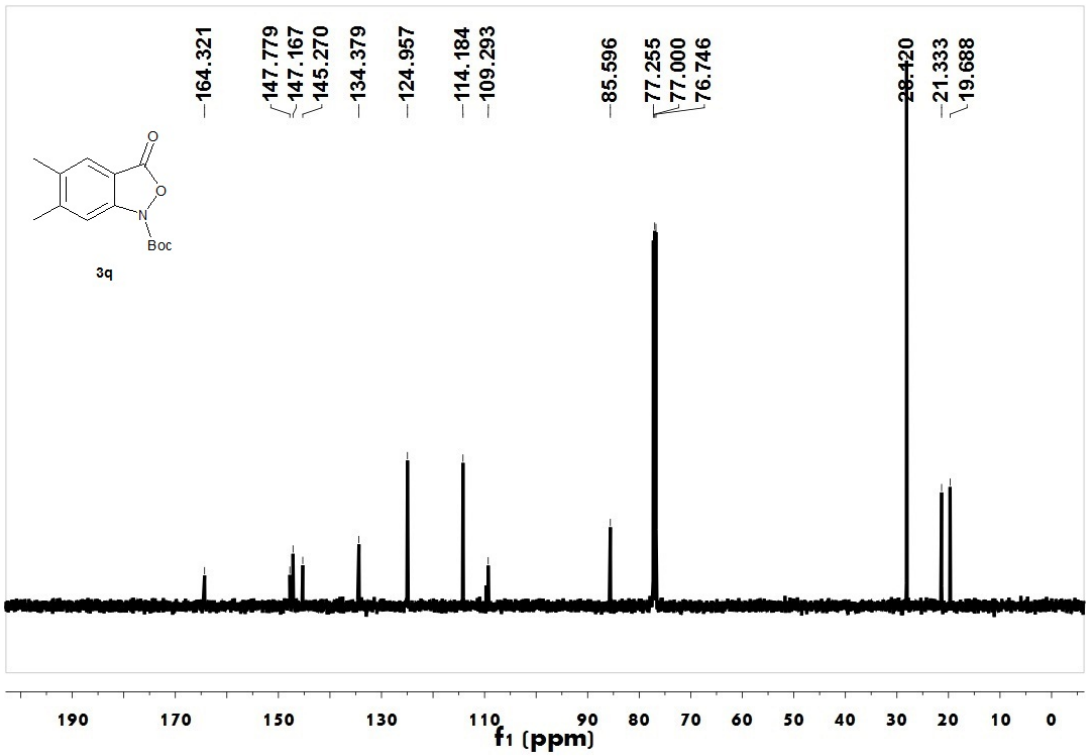

7.435
7.417
7.358
7.340
4.160
3.907
4.625
O
O
O
O
N
Boc
3o
1.00
1.04
3.01
3.20
9.18
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

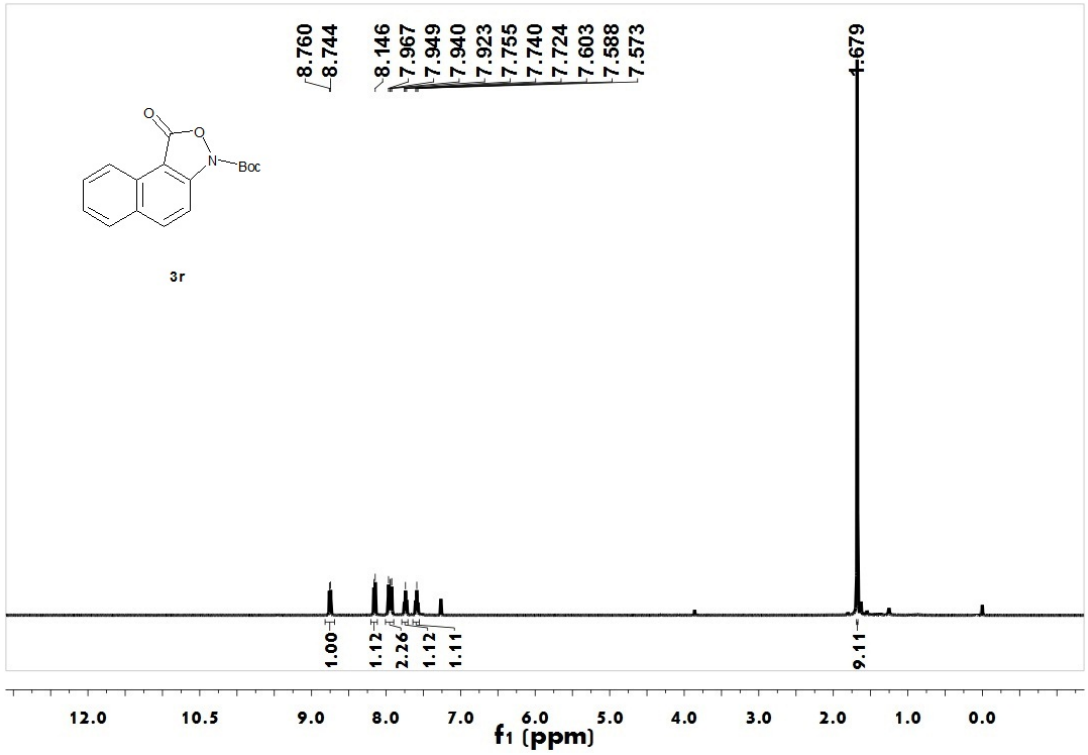

161.974
148.212
147.954
147.521
141.268
123.088
107.696
105.129
85.625
77.254
77.000
76.745
62.500
57.571
28.414
O
O
O
O
N
Boc
3o
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

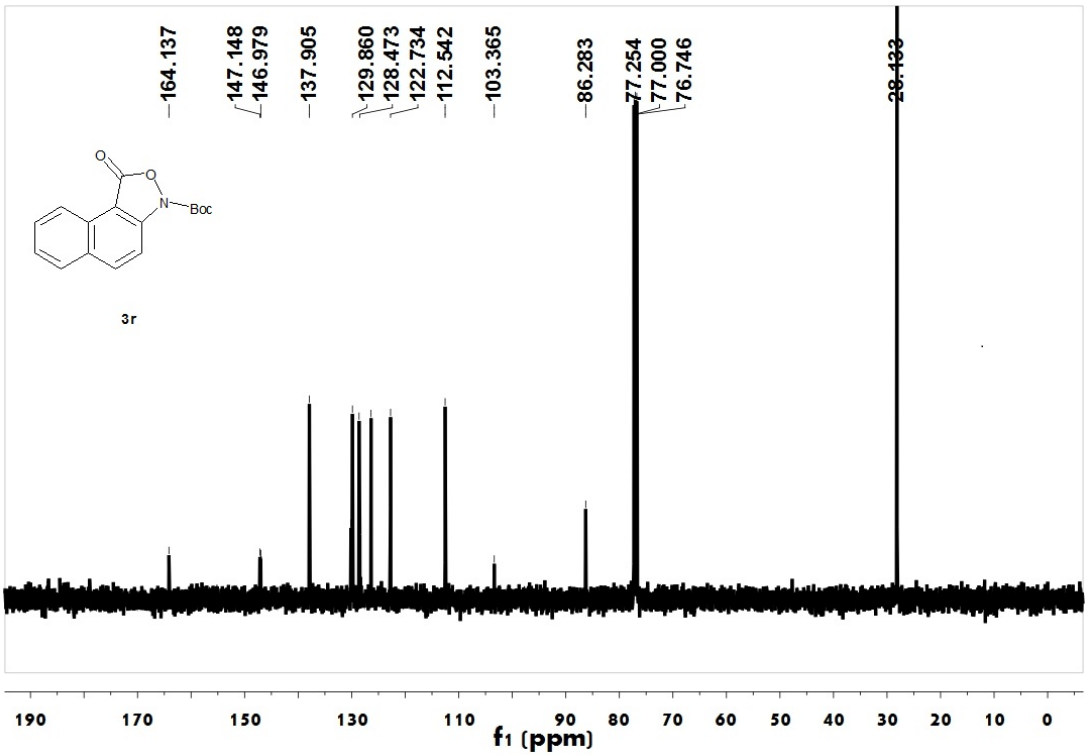

7.616
7.587
2.411
2.336
4.637
O
O
N
Boc
3p
1.00
1.05
3.10
3.08
9.18
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

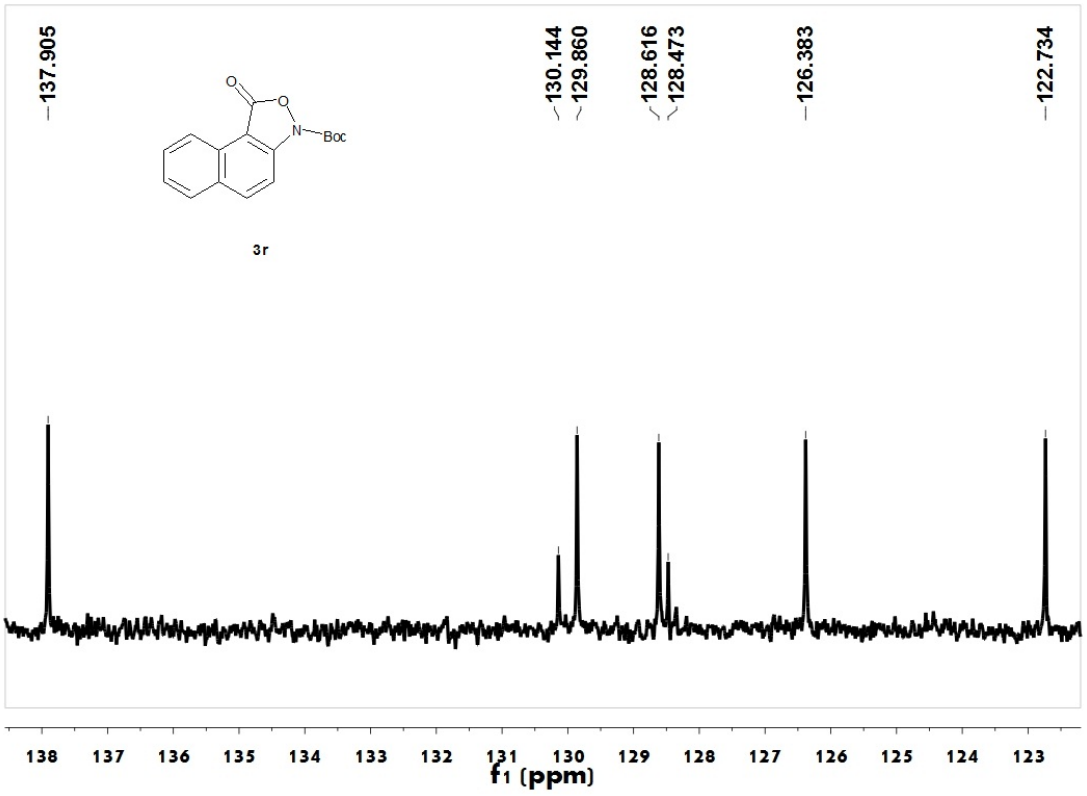

164.230
147.717
147.658
147.036
139.259
127.479
111.142
107.402
85.557
77.254
77.000
76.746
28.440
22.392
17.191
O
O
N
Boc
3p
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

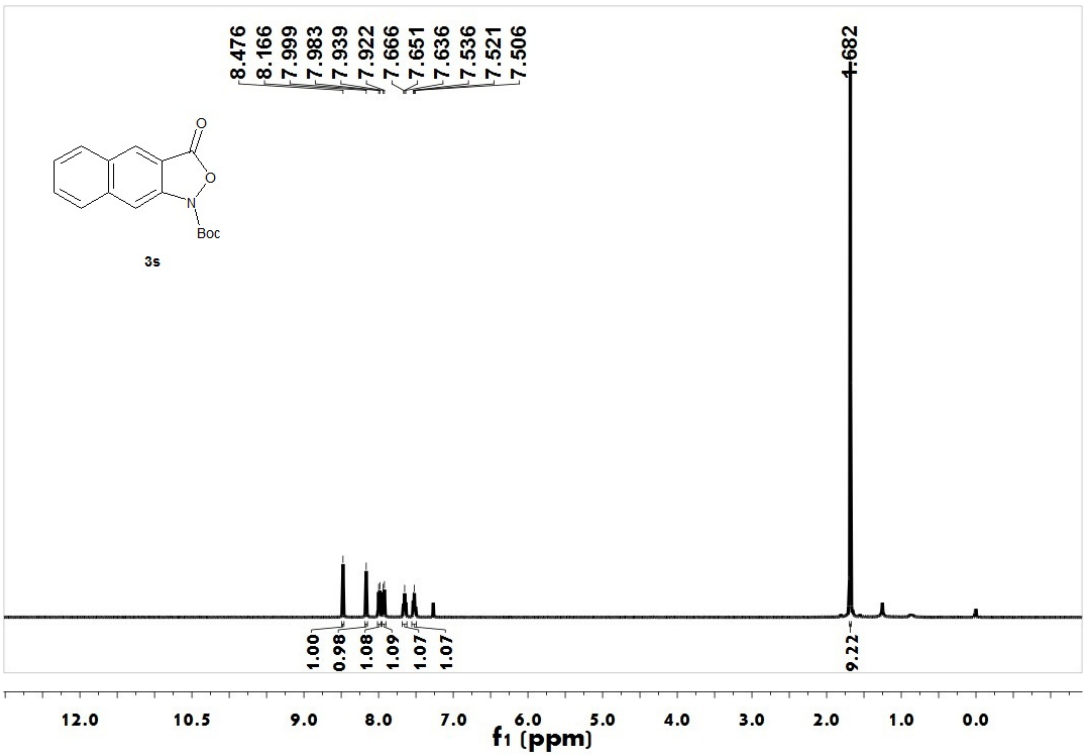

7.448
6.898
2.622
2.448
1.633
O
O
N
Boc
3q
1.00
1.03
3.13
3.09
9.26
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

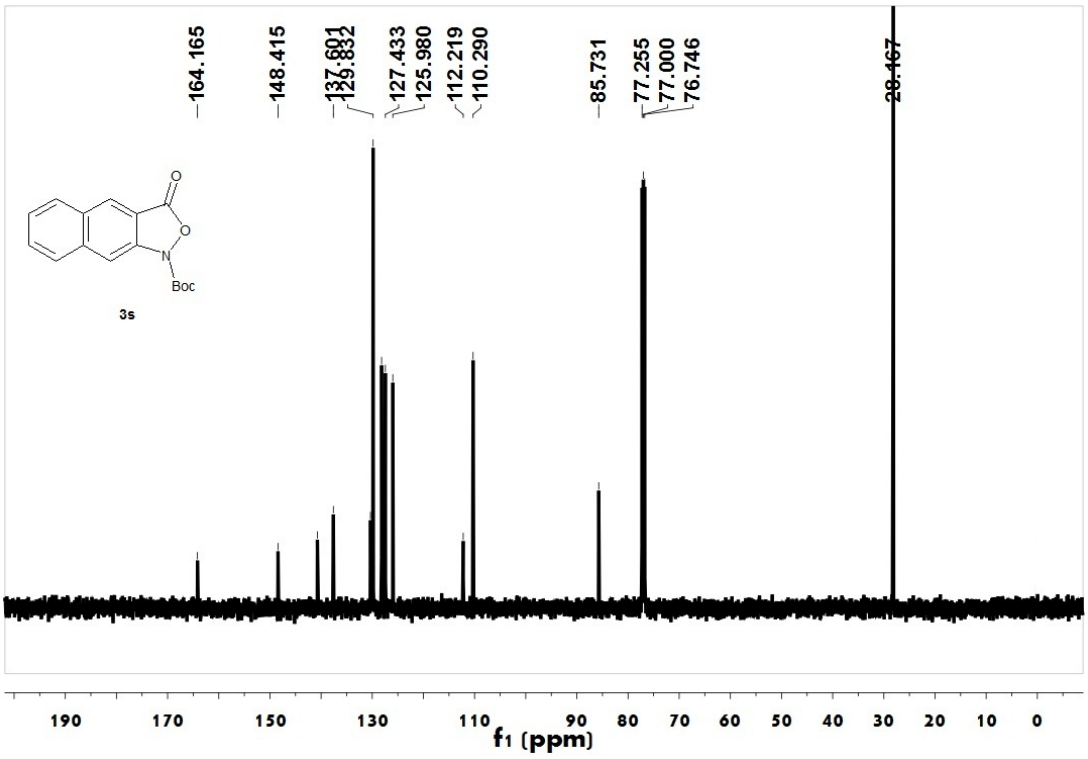

164.321
147.779
147.167
145.270
134.379
124.957
114.184
109.293
85.596
77.255
77.000
76.746
28.420
21.333
19.688
O
O
N
Boc
3q
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

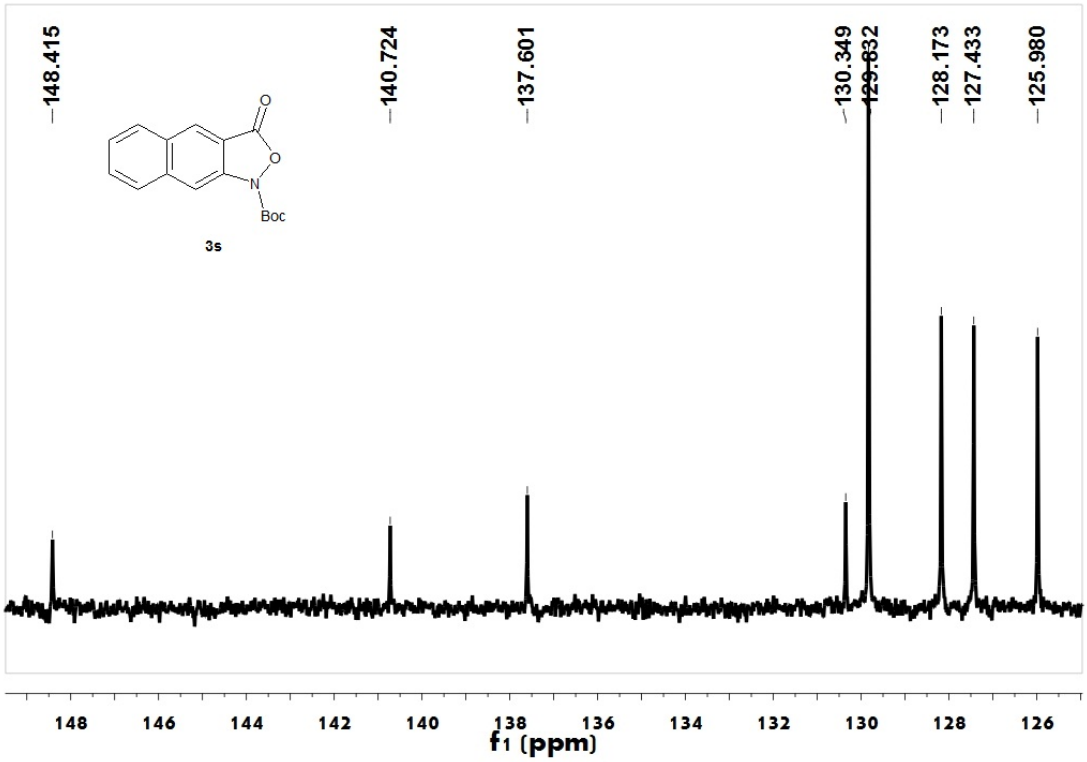

8.760
8.744
8.146
7.967
7.949
7.940
7.923
7.755
7.740
7.724
7.603
7.588
7.573
4.679
O
O
N
Boc
3r
1.00
1.12
2.26
1.12
1.11
9.11
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


164.137
147.148
146.979
137.905
129.860
128.473
122.734
112.542
103.365
86.283
77.254
77.000
76.746
28.433
O
O
N
Boc
3r
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


137.905
130.144
129.860
128.616
128.473
126.383
122.734
O
O
N
Boc
3r
138
137
136
135
134
133
132
131
130
129
128
127
126
125
124
123
f1 (ppm)


8.476
8.166
7.999
7.983
7.939
7.922
7.666
7.651
7.636
7.536
7.521
7.506
4.682
O
O
N
Boc
3s
1.00
0.98
1.08
1.09
1.07
1.07
9.22
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


164.165
148.415
137.601
129.832
127.433
125.980
112.219
110.290
85.731
77.255
77.000
76.746
28.167
O
O
N
Boc
3s
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


148.415
140.724
137.601
130.349
129.832
128.173
127.433
125.980
O
O
N
Boc
3s
148
146
144
142
140
138
136
134
132
130
128
126
f1 (ppm)